PETROLEUM REFINER

SPECIALIZED FOR THE OIL REFINING INDUSTRY, INCLUDING PETRO-CHEMICAL ENGINEERING, PETROLEUM SYNTHETICS AND NATURAL GASOLINE

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L. S. DANIELS
Associate Editor—Refining

C. C. PRYOR
Associate Editor—Cycling and Natural Gasoline

GRADY TRIPLETT
Editor

J. KENT RIDLEY
Managing Editor

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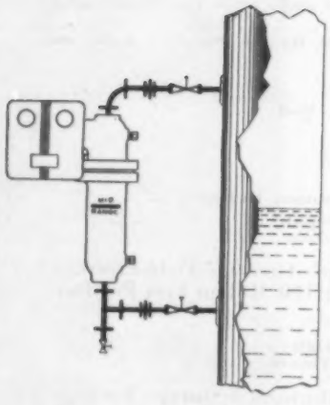

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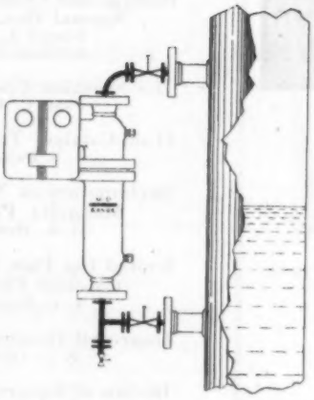

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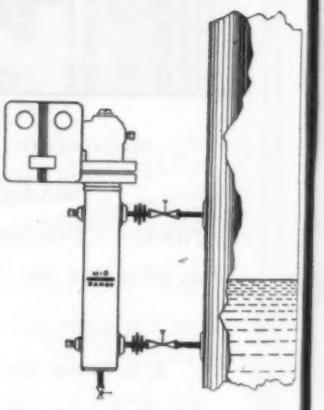

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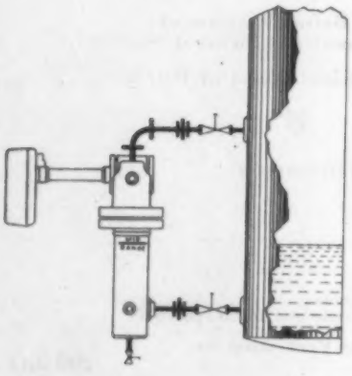

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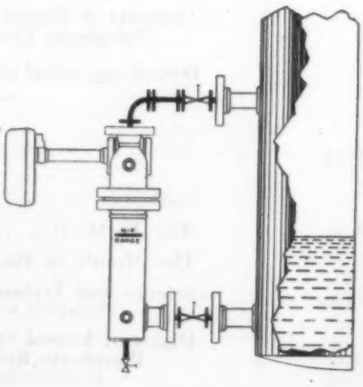

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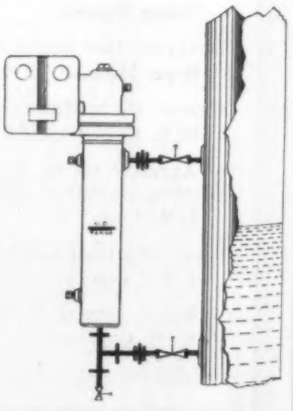

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THE LOOK BOX

THOSE inclined to look with deep approval on ventures in petro-chemical manufacture will do well to note observations on the subject from a recent study of Standard Oil Company of California. The gist of the observation is that there is a lull of several years between technical discovery and dollar profit. Some sentences from the study follow:

Long Pull

"It has been the policy of the company to establish itself in the field of intermediate chemicals where petroleum products can be converted into chemical materials at a decided price advantage. This avoids the problems inherent in the merchandising of consumers' products on the one hand and the doubtful advantage of converting petroleum products into the highly competitive field of low-priced industrial chemicals on the other. A new line of intermediate chemicals can only be introduced slowly. In each case it must be irrefutably demonstrated to the prospective customer that by adopting the proposed new intermediate chemical his own product will be materially improved or his costs reduced. This can be done only by highly trained technicians who are nearly as familiar with the manufacturing operations and products of the prospective customer as is the customer himself."

These quotations were followed by a statement of the experience of Monsanto Chemical Company where "on the average, seven years elapse between the time the research organization develops a new product with commercial possibilities and the time that product yields its first dollar of profit."

NO DEVELOPMENT better portrays the trend in refining than the status of the Petroleum Division of the American Chemical Society. It is celebrating its twenty-fifth anniversary this month, being the youngest of the eighteen professional divisions of the organization. Despite its youth, it is the largest division within the society.

Youngest and The Largest

The twenty-fifth anniversary, while taking note of this quarter century, has an eye open to the future, which some are estimating in the light of experience. Petroleum refining only recently became a chemical industry. For a long time it should hold its place within the ACS family.

IT IS heartening to realize that the State Department is giving support to the effort of our nationals to develop oil properties in foreign countries, especially the Near East. Why this should come before the American public, backed by the proposition that we face a domestic oil shortage, is questionable.

Always, the Shortage

For some reason this is the approach of most of the national efforts in oil affairs. While Harold L. Ickes was beating the air waves in behalf of more stringent federal control he based his pleadings on the proposition that this nation faces early exhaustion of its petroleum resources. Now the State Department adopts that premise as it goes forth to assist our nationals in their foreign oil affairs.

An oil shortage is no certainty. That it can come is admitted. When it will come is assumption. That it is immediate is not true. The oil

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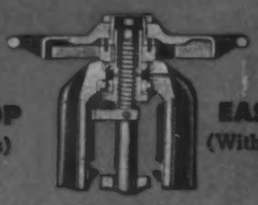

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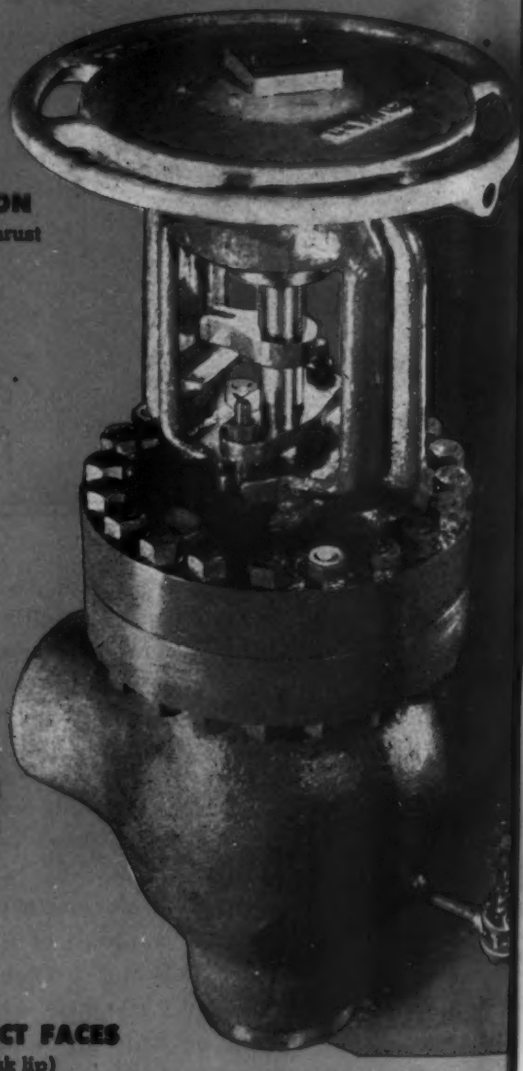

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resources within the borders of the United States are sufficient to justify the State Department in a more confident approach to its program of foreign assistance. The admission of shortage seems like weakness rather than strength.

Another questionable phase of the program lies in the tendency to look upon the Near East as a source of crude oil in case of war. That was unmistakable in the radio program sponsored by State Department representatives early in August, in which military men took part. To look upon oil from the shores of the Persian Gulf of the Mediterranean Sea as valuable in case of war is preposterous.

Oil reserves of the Caribbean area can be classed as such a reserve and it is here that both private venture and governmental assistance should look. The American interests that go into these countries, obey the laws and develop oil fields deserve the support of all department of the government of the United States. In such the industry will be doing its part to assure oil for peaceful uses in a location that has the added asset of availability in case of war.

Billion Barrels

ALONG with the announcement that the Baton Rouge plant of Standard Oil Company of New Jersey had processed its billionth barrel of crude oil in August came some interesting figures on what is involved in reaching such a total in processing. Value of this crude oil total was $1,560,000,000. Wages and salaries were $380,000,000. The State of Louisiana received $112,223,000 in taxes while the billion barrels were being processed.

This plant processed its first crude oil in November, 1909, when it had 750 employes. Now throughput is at an all-time high and the plant has 8,500 employes. Additional units are under construction and others are planned.

The Baton Rouge plant is not the first to process a billion barrels of crude oil, although such records are few. What is significant is that the trend at Baton Rouge is typical of an industry.

Good Turn

IT IS likely that the petroleum industry will respond to the suggestion of Gustav Egloff, who suggests that books, especially scientific and technical literature, be poured into China to help in rehabilitation of its libraries. If the destitute peoples ever regain the status of self sufficiency it will be by application of knowledge. China, more than any other nation, suffered from the war. For nine years it has been without literature. Along with the food and clothing that go there to sustain life, books and periodicals should be sent to support learning.

Good, If Not Perfect

IN HIS remarks before stockholders of Standard Oil Company (New Jersey), F. W. Abrams, chairman of the board, set the two divergent economic systems of the world one against the other, and gave this tribute to the one of his preference:

"We believe in a fair competitive system because it seems to us that in the long run it gives people more goods of better quality and at lower prices than they would have under any other system. This is not to say that our system is perfect; it is undoubtedly subject to improvement. But while striving to improve it we must not sacrifice its features of superiority. In our opinion the evidence is clear that people will fare better under an economy based on the fair competition of free people than under any other yet devised. The benefits which have accrued to the American people, for example, from our competitive, vigorous oil industry, both in war and in peace, are tremendous. No other nation has been so successful as we in the United States in discovering oil and bringing it to people in useful form."

Keep It Moving

EVIDENTLY it takes a long time for discarded refining equipment to find its way back to the steel mills. Only recently a drive through a refining center brought into view a junk yard whose pumps and heat-exchanger jackets looked for all the world exactly like those in place there a year ago. When its usefulness is ended in the refining centers, that scrap is needed back in the steel-making centers. To some extent, however, the shortage in scrap may be due to the shortage of finished steel products. On that same journey one engineer recited the difficulties of getting steel products and observed:

"We have worked our scrap pile until all we get now is steel filings."

Still the steel industry needs scrap and whatever can be released should be shipped and not piled in junk yards.

Future activities in research in our industry involve the development of economically practical use of gas synthesis, hydrogenation of coal and shale and exploitation of tar sands. The world still possesses great supplies of liquefied petroleum and gas and in addition has vast supplies of sedimentary solids which through the developments of research can be converted into liquid hydrocarbons in such quantity to insure supplies for many years to come.

In all this we must recognize the competitive force of other types of useful energy—certainly atomic energy is in the offing and awaiting only the development through research of means to harness it for whatever practical purpose it can service.

HENRY H. HEWETSON, *President*
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SULFUR In Light Petroleum Products

E. T. SCAFE
Socony-Vacuum Oil Company, Inc.

THE problem of sulfur in petroleum and its products is not new. The early refiners of Pennsylvania crude were faced with it, although admittedly to a small degree, and the problem has increased since that time as production frontiers moved westward and new fields were discovered. At present it is assuming increased importance because of the necessity of refining crudes of generally higher sulfur content than previously and the fear of some technicians that any general increase in the sulfur content of a petroleum product will have an adverse effect upon its performance.

This paper is intended to give some general comments on the situation, to present the results of some experimental desulfurization work on a high-sulfur crude and to make some comments on the effects of sulfur and its compounds upon the performance of petroleum products.

The paper was presented before the eleventh annual petroleum meeting, Wichita Section, American Chemical Society, Wichita, Kansas, June 6 and 7, 1946.

THE sulfur content of crude oil has a marked influence upon the sulfur content of the finished petroleum products. Let us, then, examine the sulfur contents of the crude production in this country. The data in Figure 1 show the range of sulfur content of crudes by states. These data were obtained from the numerous publications of the Bureau of Mines on crude analysis. Since only the data on a fraction of the producing wells have been published, it is highly probable that some must fall outside this range. Nevertheless, for practical purposes we may assume that these values are fairly representative.

It may be noted that of the 19 oil producing states listed three states have some crudes in excess of 3 percent-weight sulfur; two states have some crudes in excess of 2, but less than 3 percent-weight sulfur; five states have some crudes in excess of 1, but less than 2 percent-weight sulfur; three states have some crudes in excess of 0.5, but less than 1 percent-weight sulfur, and six states have some crudes with maximum sulfur content below 0.5 percent-weight.

With the exception of Pennsylvania, New York, West Virginia, Kentucky, Illinois, Indiana and Colorado, all states appear to be plagued with crudes which fall in the category of high-sulfur crudes. It is significant that the areas of high-sulfur crudes are dispersed throughout the United States and are not centralized in any one locality or section.

While Figure 1 does not give a weighted average of sulfur content based on volume of crude produced, it is generally recognized that the availability of the low-sulfur crudes is diminishing, which forces the refiner to process the higher-sulfur crudes, with the net result that the average overall sulfur content of the crudes being processed is increasing.

The exigencies of wartime production of petroleum products were largely responsible for accelerating the processing of high-sulfur crudes. Conservation of natural resources will be an important consideration in the continued use of these crudes.

The problem of handling high-sulfur crudes has been approached differently by the various petroleum companies. Some have made the decision to convert entire refineries over to processing high-sulfur crudes; others have segregated the stock and processed it separately in certain units, while others have chosen to blend the high-sulfur with low-sulfur crudes and process the mixture. Wartime experience indicated that up to 5 percent-volume of high-sulfur crudes could be blended with Mid-Continent pipe line oil without imposing any great hardships upon the refineries. Of the schemes mentioned, the conversion of entire refineries or sections thereof over to processing high-sulfur crudes appears to have found the most favor among the major oil companies. At present, one Midwest refinery is processing 35,000 barrels daily of crude of 1.5 percent-weight sulfur.

FIGURE 1
Range of sulfur contents of crude oil by states.

West Texas Crudes (Slaughter-Duggan)

The West Texas crudes have achieved some notoriety by virtue of their sulfur content. A tabulation of 62 crudes was given in the *Oil and Gas Journal* by W. L. Nelson and is reproduced in Table 1. It is of interest to note that 8 of the 62 crudes listed for this area noted for high-sulfur crudes have sulfur contents below 0.5 percent-weight.

The highest sulfur content listed is 4.17 percent-weight for Fuhrman Crude (No. 35).

Included in this list of crudes is Slaughter-Duggan which has been used by Socony-Vacuum Oil Company's research and development laboratories at Paulsboro as a representative high-sulfur

crude. You will note it listed as No. 19 and see that it contains 2 percent-weight sulfur. The specific samples of crude received had a sulfur content of 2.1 percent-weight which is in close agreement with the published data. A distillation of this crude yielded the products shown in Table 2.

For a more complete insight into the distribution of the sulfur let us examine the sulfur material balance and the estimated volume percentage of the products represented by this sulfur, which are shown in Table 3.

Eighty-eight percent of the sulfur present in this crude is contained in the gasoil and residual fractions upon primary distillation. The gasoline and naphtha fractions contain only 6 percent of the total sulfur. Nevertheless this amount is sufficiently high to be troublesome. The last column in this table shows the estimated volume percent of the product represented by the sulfur-bearing compounds. These values are significant since they represent the minimum loss of product to be expected by desulfurization processes based upon complete elimination of the sulfur-bearing compounds by solvent-extraction or similar processes.

It may be noted in this table that 47 percent-

TABLE 1

Properties of Some West Texas Crude Oils

No. or Symbol	NAME OF CRUDE	API	Sulfur Percent Weight
1	Big Lake	38.2	0.36
2	Chalk	32.1	0.84
3	Chalk	31.3	0.91
4	Chalk	26.8	2.02
5	Church and Fields	31.1	2.69
6	McElroy	33.0	2.42
7	McElroy	32.8	2.40
8	McCamey	24.5	2.29
9	McCamey	25.6	2.23
10	Westbrook	25.8	....
11	World	29.7	0.59
12	Hendricks	30.0	1.53
13	Yates	30.0	1.61
14	Yates	30.4	....
15	Yates	29.3	2.40
16	Yates	29.6	1.55
17	Texon (nearly paraffin base)	38.2	0.26
18	Westbrook	29.2	1.09
19	Slaughter-Duggan	31.6	2.00
20	Wasson	33.5	1.66
21	Slaughter	31.7	1.93
22	Winkler County	28.0	....
23	Winkler County	36.2	....
24	Taylor-Link, Pecos County	30.2	....
25	Upton County	27.2	....
26	Pecos County	30.5	....
27	Apco	38.6	0.27
28	Big Lake	36.4	0.36
29	Big Lake	41.5	0.14
30	Cowden, Northfield	35.0	1.66
31	Crane, Cowden	32.3	2.37
32	Dune	34.4	1.49
33	Embar-Ellenburger	44.9	0.18
34	Foster	36.4	1.07
35	Fuhrman	22.5	4.17
36	Fullerton	43.2	0.48
37	Goldsmith	34.2	1.90
38	Harper	36.0	1.15
39	Hendrick	29.7	1.49
40	Howard-Glasscock	29.1	1.64
41	Iatan, East Howard	30.8	1.41
42	Jordon	34.6	2.13
43	Kermit	28.4	1.45
44	Keystone	35.2	0.84
45	Leck	29.1	1.64
46	McElroy	31.3	2.39
47	Payton	33.0	1.10
48	Pecos Valley	34.6	1.09
49	Pecos Valley	29.7	1.67
50	Seminole	35.6	1.76
51	Scarborough	36.6	1.02
52	Shipley, Silurian	30.6	0.29
53	Slaughter	32.3	1.93
54	Snyder	29.9	1.68
55	Taylor-Link	30.2	1.32
56	Tobarg	25.7	1.93
57	Waddell	44.7	0.22
58	Ward	35.0	1.77
59	Ward, South	37.2	1.27
60	Wasson	34.6	1.63
61	Wheat	38.0	0.21
62	World	29.1	0.73

TABLE 2

Yield and Properties of Products from Slaughter-Duggan Crudes

PRODUCT	Yield Percent Volume	Gravity, ° API	Sulfur, Percent Weight
Light Straight Run, 286 °F. EP	15.2	62.7	0.19
Naphtha, 280-404 °F	19.1	47.0	0.62
Light Gasoil, 437-739 °F	31.4	30.6	1.76
Heavy Gasoil*	10.5	28.0	2.68
Residuum	21.5	8.2	4.15
Total	97.7	.....	....
Crude	100.0	31.2	2.10

* 90 percent at 925 °F. by vacuum assay.

TABLE 3

Sulfur Material Balance

Basis: 100 gallons Slaughter Duggan crude

PRODUCT	Lbs.	Lbs. of Sulfur	Percent Weight of Total Sulfur	Sulfur Compounds Percent Volume*	
				of Product	of Crude
Crude	720	15.1	100	....	18.5
Light Straight Run	99	0.2	1.3	0.5	0.1
Naphtha	126	0.7	4.5	2.3	0.4
Light Gasoil	228	4.0	26.6	11	3.4
Heavy Gasoil	81	2.2	14.6	29	3.0
Residuum	174	7.1	47.0	54	11.6
Gas and Unacc't.	12	0.9	6.0	....	....
	720	15.1	100.0	....	....

* On basis that compounds are monotomic sulfur compounds

$$\frac{\text{Sp. Grav of HC's}}{\text{Sp. Grav. of S Compound}} = 0.87$$

weight of the sulfur may be concentrated in the residuum by a simple distillation procedure. This procedure is an effective means for getting rid of relatively large amounts of sulfur, particularly if the residuum is used as fuel, road oil or asphalt. Extensive studies have been made by various investigators on the reduction of sulfur content of gasolines, naphthas, kerosines and gasoils. It is not intended to give a review of this published work, but to confine the discussion to some of the experimental work conducted at our laboratory on the fractions of this Slaughter-Duggan crude.

General Types of Desulfurization Processes

Two approaches to the desulfurization problem usually are considered; the one is based upon the elimination of the sulfur as hydrogen sulfide generally by use of cold catalyst; and the other, is based upon concentrating the compounds containing the sulfur by solvent action. A third approach, based on chemically reacting the sulfur compounds with a reagent with subsequent removal of the reaction product, has been successfully applied in certain specific cases. Of these approaches, the ideal development will be that one which will eliminate the sulfur as hydrogen sulfide under mild conditions without the need of frequent regeneration of catalyst. To add one more restrictive condition, this ideal process should be simple and inexpensive so that the operation could be conducted in the oil fields, thereby removing this menace of sulfur from the refinery.

Desulfurization of Slaughter-Duggan Gasoil with Solid Catalyst

Reduction of the sulfur content of the light gasoil fraction was the objective of one phase of the work with Slaughter-Duggan crude. A summary of the results with experimental solid catalysts is given in Table 4. These solid catalysts were essentially metal

TABLE 4

Desulfurization of Slaughter-Duggan Gas Oil of 1.6% Weight Sulfur Content with Experimental Catalysts

ADDED METAL OXIDE	Sulfur Content (Percent Weight) of Product Obtained with Metal Oxide Deposited on		
	Silica	Alumina	Aluminum Silicate
None	1.0	0.9	0.5
Beryllium Oxide	0.8	....	0.6
Cadmium Oxide*	0.8	0.6	0.4
Calcium Oxide	....	....	0.4
Chromium Oxide	0.7	....	0.5
Cobalt Oxide	....	0.5	....
Ferric Oxide*	....	0.6	0.4
Magnesium Oxide	0.9	0.6	0.4
Nickel Oxide	....	0.5	0.5
Sodium Oxide	....	....	1.0
Zinc Oxide	0.6	....	0.3
Cobalt Molybdate	....	0.8	....

*Difficulty encountered in Regeneration.
Operating Conditions: Temperature 650 °F.
Space Velocity, 1 vol. of oil/vol. of catalyst/hour.

oxides on either silica, alumina or aluminosilicate carrier.

The significant observations to make from these data are as follows:

1. That an alumniosilicate cracking catalyst without any oxide promoter is one of the most effective desulfurization catalysts. Under optimum conditions, a yield of over 90 percent-volume of product was obtained.
2. Zinc oxide-type catalyst appears to give the lowest sulfur content product of the catalysts tested. This more or less confirms the published data that zinc oxide is effective for removing sulfur from hydrocarbons.
3. The most effective carrier for the metallic oxides is one with cracking activity, that is, the aluminosilicate cracking catalyst. This fact suggests that some of the sulfur in petroleum is held rather firmly in the compound and requires severe treatment to dislodge it. It further indicates that an ideal catalyst will have to be one of extremely high and specific activity.

Desulfurization of Slaughter-Duggan Gasoil with Anhydrous Hydrogen Fluoride

The second approach on reduction of sulfur content of the light gasoil was made by a solvent-extraction method using liquid anhydrous hydrogen fluoride as the solvent. A summary of these data is presented in Figure 2. These data show a 70 percent sulfur reduction with a yield of 85 percent-volume of product of 0.5 percent-weight sulfur is possible with this solvent. It is of interest to note that in both the solid catalyst and solvent-extraction studies, the minimum sulfur content obtained is approaching a limiting value in the region of 0.4 or 0.3 percent-weight sulfur. This fact may also be interpreted to mean that this gasoil contains a certain amount of sulfur compounds which are very resistant to removal or decomposition.

Combination of HF Desulfurization and Catalytic Cracking

The combination of solvent extraction with anhydrous hydrogen fluoride with the desulfurization attendant with catalytic cracking of the partially desulfurized gasoil provides a possible means of producing low-sulfur gasoline from Slaughter-Duggan crude. The results of an experiment of this nature is given in Table 5. In the catalytic-cracking phase of this experiment, the conditions were adjusted to give essentially the same coke deposition for both the untreated and HF-treated gasoils.

The desulfurization with hydrogen fluoride prior to catalytic cracking results in improved yields of motor gasoline based on the charge to cracking. Based on the untreated gasoil, however, a yield of 45.5 percent-volume is obtained, or a net loss of 2.6 percent-volume. The latter is due to the 12 percent-volume loss in cracking stock occasioned by HF treating.

The quality of products from cracking the partially desulfurized gasoil is improved over that of the products from cracking the untreated gasoil, as exemplified by the lower sulfur content and higher octane

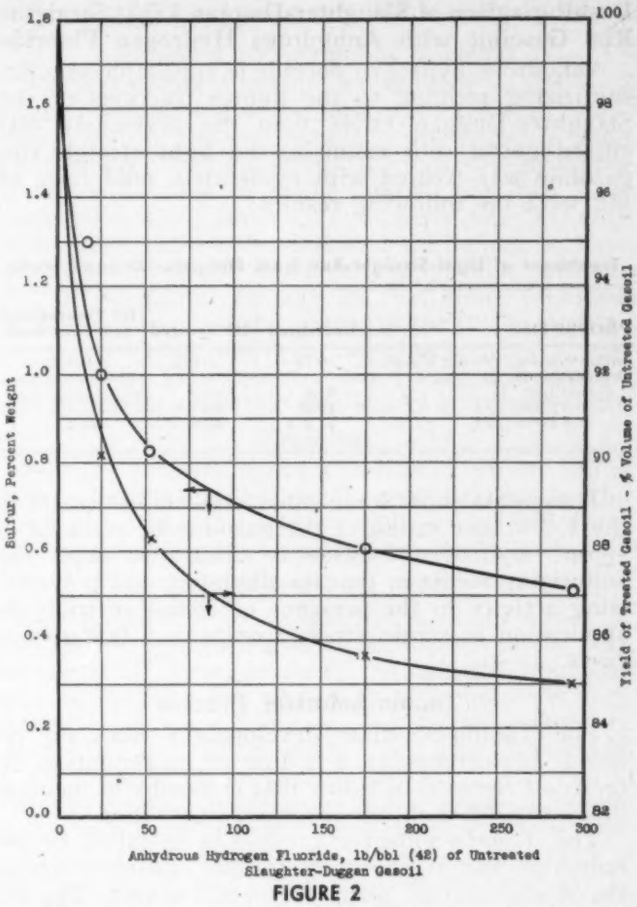

FIGURE 2

Yields and sulfur contents of Slaughter-Duggan gasoil treated with hydrogen fluoride.

rating of the gasoline. The sulfur appearing in the motor gasoline from the HF-treated gasoil is less than 4 percent of that in the original gasoil and sulfur in the motor gasoline from the untreated gasoil is less than 11 percent of that in the original gasoil. These values are significantly low upon realization that the comparable value from vapor-phase thermal cracking of the same stock is 25 percent.

The sulfur content of the cycle stocks from these two operations show an interesting behavior. In the

TABLE 5

Yield and Properties of Motor Gasoline Produced from Partially Desulfurized Slaughter-Duggan Gas Oil

	Untreated Gasoil	HL Treated Gasoil*
Charge Stock Sulphur Content, Percent Weight	1.71	0.81
Yields, Percent Volume of Charge to Cracking:		
Motor Gasoline, 10 lbs. R.V.P.	48.1	51.6
Cycle Stock	44.8	36.3
Excess Butanes	6.7	11.6
Properties:		
Motor Gasoline:		
Sulfur Content, Percent Weight	0.14	0.048
CFRM—Clear	72.8	77.5
+3.0 cc TEL	81.7	85.9
CFRR—Clear	78.3	83.9
+3.0 cc TEL	88.9	93.9
Cycle Stock:		
Sulfur Content, Percent Weight	1.37	1.18

* Represent 88% Vol. of untreated gasoil.

cycle stock from the untreated gasoil, the sulfur content was lower than the feed stock, while in the cycle stock from the HF-treated gasoil, the sulfur content was appreciably higher than the feed stock. This example is another illustration of the stability of some types of sulfur compounds.

Desulfurization of Slaughter-Duggan Light Straight-Run Gasoline with Anhydrous Hydrogen Fluoride

Anhydrous hydrogen fluoride is applicable as a desulfurizing medium to the lighter fractions of the Slaughter-Duggan crude than the previously discussed gasoil. For example, the light straight-run gasoline was treated with moderately mild ratio of HF with the following results:

Treatment of Light Straight-Run from Slaughter-Duggan Crude

PROPERTIES	Untreated	HF Treated	HF Treated and Isopentanized
Sulfur Content, Percent Weight.	0.19	0.012	0.015
Octane Numbers:			
F-3—Clear.....	64.4	65.3	.....
+3.0 cc TEL.....	76.0	86.1	.....
+4.0 cc TEL.....	77.7	87.9	91.2

These data show some substantial improvement in the F-3 octane rating of the gasoline by removal of sulfur. Hydrogen fluoride is similar to other desulfurizing media in that its alkylating and polymerizing activity in the presence of olefins restricts its application to virgin stocks except in a few special cases.

Tannin-Solutizer Process

The Tannin-Solutizer development work for removal of mercaptans is a line of investigation directed at removal of sulfur that is worthy of mention in connection with our laboratory activities.

The Tannin-Solutizer process is installed in ten refineries and is receiving serious consideration in the modernization plans of several others. The extraction stage of this process uses a solutizer solution of sodium or potassium cresylate or isobutyrate, and the regenration stage employs tannin as catalyst for the air oxidation of the mercaptan to disulfides. The latter products are settled and/or washed from the solution to allow effective reduction of the sulfur content of the hydrocarbon product. The major advantages to the process lie in its applicability to a wide range of mercaptan-content gasolines, including those from high-sulfur crudes, and the low temperature of operation of the regenerative step. This latter item is important from the equipment-corrosion standpoint, as those who operate steam regenerative processes of this type know.

The effective removal of 95 percent of the mercaptan sulfur from gasoline is possible with this process.

Effect of Sulfur Upon Performance

The foregoing remarks conclude the prepared comments on the general subject of desulfurization and lead us to the next topic, which is concerned with the *Effect of Sulfur and Its Compounds upon the Performance of Petroleum Products.* We will, of necessity, tread lightly on this subject because many of the facts are unknown. This situation may be traced to the probable fact that the investigators have been occupied with the quality items which have more significance on performance than sulfur and that any comprehensive study would involve considerable time and money by virtue of the many ramifications to the problem.

In the most optimistic sense we can only say that we have little technical information to support the present sulfur limitations in our specifications. These limitations generally attempt to minimize the effect of sulfur in two respects:

a. Such corrosion of material in contact with liquid products as may be imparted by elemental sulfur and hydrogen sulfide. The criterion for control of this item is the absence of corrosion to copper or other numerous metals and materials.

b. The performance of products with respect to corrosion of materials, wear, engine deposits which may be affected by the products of combustion of sulfur compounds. The criterion in the specification for this control is the total sulfur content of the product.

A third limitation aimed at controlling the mercaptan-sulfur content below any significant level is included in specifications of some products. This item is considered outside the scope of our discussion, since it affects sales performance to a greater extent than field performance.

Corrosive Sulfur

The first item, that is, the limitation on corrosive sulfur and hydrogen sulfide, may be disposed of rather briefly. It is generally specified that light products be free of corrosive sulfur and sulfur compounds as indicated by the absence of corrosion on a copper specimen or other sensitive material under accelerated test conditions. This specification item probably originated with the use of the doctor-treating process as a control test against the use of excessive sulfur during treating. Nevertheless, it has remained, and with an increase in severity of test conditions and a more critical examination of the degree of discoloration or corrosion being exercised by the inspector, it has become a specification which has, at times, caused the refiner some concern over its exact significance. It is doubtful that trace amounts of sulfur have any significant effect upon the corrosion of material with which it comes into contact, notwithstanding the infrequent complaints reported to be caused by sulfur.

Over a period of years we have heard of occasional instances in which products showing evidence of corrosion to copper have corroded lines in domestic fuel-oil and kerosine service sufficiently to cause failure, of instances in which the failure of flow regulators and float controls was blamed on corrosive sulfur, and of another instance in which the discoloration of a sensitive paint pigment, chrome yellow, was reported to be due to corrosive sulfur. The isolated nature of these instances, and the lack of evidence, has left considerable doubt as to the true responsibility for these failures.

During the war, the copper-corrosion characteristics of some aviation-gasoline production as determined by the copper-dish method reached a critical stage. This trouble was a combination of circumstances involving interpretation of the method, complications caused by additives in the gasoline, and other problems associated with expanded production. These problems were overcome and subsequent production was satisfactory.

It is to be emphasized that this problem was concerned with only the interpretation of specifications and not with the performance of the fuel in service. Inquiries directed toward finding out the history of any cases of failure or corrosion attributable to

elemental sulfur in aviation gasoline failed to produce any recorded instances.

The present situation with the corrosive-sulfur specifications designating a submerged-copper-strip specimen is generally satisfactory. It is neither too restrictive with respect to the ability of the refiners to conform to it, nor does it permit any significant amounts of sulfur to remain in the marketable products. However, three suggestions are offered which would place the specification on a firmer basis from a technical standpoint.

1. Develop a precise quantitative test for sulfur or sulfide corrosion in order to eliminate the present qualitative one which is frequently used in a quantitative manner by expressing the degree of discoloration in terms of sulfur concentration. This latter technique may be misleading because it does not consider the depth of corrosion on the copper specimen.
2. Write the specifications in terms of permissible elemental sulfur content instead of its behavior on a polished copper specimen. This implies that minute traces will be tolerated in the finished products and only those in contact with delicate and easily corroded materials will be specified "free of sulfur."
3. Standardize on the quantitative test and retire the numerous versions of the copper-corrosion tests of which ASTM sponsors no less than six: the mercury, silver, bronze powder, lead carbonate and similar tests.

Sulfur Compounds and Combustion Products

In considering the second item, that is, the effect of sulfur compounds and their combustion products upon performance of finished petroleum products, it is necessary to refer to specific products instead of considering all products in one group as was possible in the case of corrosive sulfur. Furthermore, it will be expedient for our discussion to classify these products into two arbitrary groups.

The first classification is predicated on the basis that normal processing of these products for quality considerations other than sulfur content usually will be sufficient for controlling the sulfur content below any critical performance limit. Nevertheless, specific problems with these products may arise if high-sulfur crudes are the only stock for the production. In this first group are such products as

PRODUCT	Typical Specifications
Liquefied Petroleum Gas	15 gr/100 cu. ft.
Aviation Gasoline	0.05
Aliphatic and Aromatic Solvents	0.05
Kerosine	0.12
Domestic Fuel	No limit

Liquefied Petroleum Gas

The obnoxious sulfur derivatives in the LPG products are hydrogen sulfide and elemental sulfur. The effect of these compounds on the delicate control instruments in municipal and domestic gas installations and their combustion products in heat-treating applications are reported to be the basis for the above-mentioned specification limits.

We have somewhat of a paradoxical situation with this product in that it is one in which sulfur compounds have to be added as an odorant for safety purposes.

Modern refining techniques in use for preparing polymerization and alkylation feed stock is capable of controlling the liquefied petroleum gas within the specification limits and no particular problems are anticipated with these products.

Aviation Gasolines

The record of production of aviation gasolines with respect to sulfur has been so low as to preclude any opportunity to study its effect upon performance. Moreover, the practice of making exacting specifications for aviation fuels and the economic factors involved in obtaining the maximum antiknock improvement for the TEL added is so firmly established on the safe side that it is unlikely that sulfur specifications will ever be liberalized to a point where it will be a serious problem with this product.

Aliphatic and Aromatic Solvents

The petroleum solvents are another example of products whose sulfur specifications have been established well on the safe side in order to insure good performance, and consequently, recorded instances of difficulties due to sulfur content are rare. This tendency toward low-sulfur solvents has probably been influenced by the desire of the refiner to have products which are universally applicable without fear of complaint. It may be that some problems with sulfur may be encountered in the future application of solvents and will cause a revision of the specification. However, for the present, it appears that the limits are firmly established and changes are

Kerosine

Burning kerosine appears to have a critical sulfur limit with respect to chimney cleanliness in the range of 0.1 to 0.15 percent-weight which will probably continue to be a controlling factor with this

Domestic Fuel Oil

The absence of sulfur specifications for domestic fuel oil denotes that sulfur is not critical in the performance of these products. In certain industrial uses of distillate fuel oils, such as in heat treating, ceramic and glass furnaces, the sulfur content is reported to be a critical factor and is usually specified in the range of 0.5 percent-weight.

In the second classification are products in which the present level of sulfur contents is reported to be a factor in performance, or at least approaching a critical limit. In this group are such products as:

Product	Range of Specifications
Motor Gasoline	0.1—0.25
Diesel Fuel	0.5—1.0

Motor Gasoline

The industry, with the possible exception of the West Coast section, has been guided by the federal and state specification of 0.1 percent-weight sulfur for motor gasoline. This specification was reported to be an outgrowth of an incident in which cars in government service during the summer season and stored during the winter season were reported to have exhibited appreciable wear and corrosion of the wrist pins which was attributed to the sulfur content of the fuel.

During the war it became necessary to review the sulfur specification of 0.1 percent-weight because it was restricting production of 80-octane all-purpose motor fuel. A review of the meagre technical information failed to reveal any basis for limiting the sulfur content to 0.1 percent-weight so it was recommended to increase the limit to 0.25 percent-weight sulfur on the basis that gasolines of this and higher sulfur contents had been marketed on the West Coast without any discernible harmful effects. Consideration of a second increase to 0.4 percent-weight sulfur was pending at the termination of the war. In the absence of service complaints from 80-octane

motor gasoline of 0.25 percent-weight sulfur, we may assume that its effects were not noticeable, or, if present, were inconsequential.

Some of the reported effects in which sulfur compounds or its acidic combustion product may have an effect upon performance of the automotive engine are: (1) deposits in induction system by virtue of an increase in gum formation imparted by some sulfur compounds; (2) the occurrence of pre-ignition with leaded gasolines; (3) corrosion or wear of moving parts, and (4) corrosion of the exhaust system where condensation may occur.

The common belief is that the oxides of sulfur resulting from combustion are responsible for any ill effects of sulfur-containing fuel. An attempt was made in our laboratory to analyze the exhaust gases from several fuels of varying sulfur content. For this purpose a thermal gasoline containing 0.1, 0.5 and 1.0 percent-weight of added sulfur in the form of a thiophene derivative was used. Calculations showed that the amount of oxides of sulfur would be very small so an elaborate sampling system allowing the sample to be taken and maintained under subatmospheric pressure in order to keep the moisture in vapor form, and the sensitive mass spectrograph was employed for the analyses work. The analytical results confirmed our suspicions that the concentrations would be too small to be detected, but it did serve to emphasize that the effect of the oxidation products of sulfur is probably an accumulative effect, since the products are present in such a dilute mixture.

In the present stage of analytical reasoning, two schools of thought have been advanced on the general sulfur problem. Some investigators believe that it is only the sulfur content which needs to be considered, while others believe that the type of compound containing the sulphur is important. If the latter line is the significant one, this additional complication will possibly retard the solution of the problem.

Diesel Fuel

The problem of sulfur in diesel fuels, particularly those for high speed service, is probably receiving the most attention from a technical viewpoint at the present time. Diesel fuels are not desulfurized to the same extent as gasoline in the basic distillation and/or cracking process, and, consequently, have an appreciably higher level of sulfur content.

The particular concern with diesel fuels is the excessive wear of certain moving parts, and engine deposits reported to be caused by sulfur. The published data of Blackwood and Cloud (SAE Journal, Vol. 51, 1943) and other independent data indicate that in the range of sulfur content of 0.2 to 1.3 percent-weight, overall engine fouling, piston ring belt and skirt deposits, top piston ring and top cylinder wear appear to be related to sulfur content, while cooperative studies on two fuels of 0.2 and 0.8 percent-weight sulfur by five laboratories were inconclusive in respect to the effect of sulfur. Of interest in connection with these data is that variations in type of engine and load condition produced greater variations in fouling and wear than observed between widely varying sulfur-content fuels under a given condition.

The Coordinating Research Council in preparing a *Summary of Engine Performance as Related to Fuel Properties (Page 309, 1946 Handbook)*, did not consider the available data conclusive with respect to the effect of sulfur and reported that no independent data were available. Nevertheless, this organization recognizes the problem as one of vital importance and have sponsored a group on the Sulfur Limit of Diesel Fuel which has the objective of developing technical information on this subject.

Types of Sulfur Compounds in Diesel Fuel

We have had an occasion in our laboratory to analyze five diesel fuels varying in sulfur content from 0.2 to 1.2 percent-weight for types of sulfur compounds present. The results of the first attempt at this complicated problem by employing the technique developed and reported by Ball of the Bureau of Mines for gasoline was hardly satisfactory because only 25 to 39 percent of the sulfur compounds were identified. (Note: Ball did not recommend a method for products boiling above approximately 500° F.) Nevertheless, the results may be of interest to this group. For the fuels tested, the sulfur compounds, reported as percent-weight sulfur, were as follows:

SULFUR COMPOUND	Range of Sulfur Content Percent-Weight
Hydrogen Sulfide	0.000—0.000
Mercaptans	0.001—0.036
Free Sulfur	0.000—0.030
Disulfides	0.005—0.059
Aliphatic Sulfides	0.020—0.144
Aromatic Sulfides and Thiophenes	0.030—0.170
Water-Soluble Sulfur Compounds	0.000—0.000
Residual Sulfur	0.125—0.914
Sulfur Identified	0.085—0.359
Total Sulfur	0.21 —1.25

It is apparent from this work that some test method development work will be required before an exhaustive study of effect of sulfur compounds upon performance can be made.

Gas Turbine Fuels

In anticipation of a question concerning the effect of sulfur on performance of gas turbine and jet fuel, it may be stated that little information is available on this subject. The effect of sulfur in this type fuel may be manifested in the same manner as other fuels, i.e., combustion chamber deposits and corrosion from combustion products. We have some experimental information which shows that some types of sulfur compounds reduce deposit formation in jet burners. Corrosion from oxides of sulfur at the high temperatures encountered has not been exhaustively studied. However, preliminary studies indicate that present materials of construction are not readily attacked by the oxides at high temperatures.

In closing, it is apparent that we have only meagre information on the effect of sulfur content of the product upon its performance. It is also apparent that it will take a comprehensive study and experimental program to elucidate all the facts so that logical specifications may be developed on the basis of technical information rather than a consensus of the producing or consuming industries. Some cooperative experimental work along these lines is in progress under the auspices of the Coordinating Research Council. It is hoped that this program will have the support of all interested groups and that the programs will be expanded to give us a comprehensive view of the relationship between sulfur and performance. It is fairly certain that the petroleum industry will accept the technical facts and control the sulfur content of their products to gain the advantage of any reasonable increase in performance.

Boiling and Condensing Film Coefficients For Water and Normal Hexane

DONALD L. KATZ
Professor of Chemical Engineering, University of Michigan

GEORGE H. HANSON
Phillips Petroleum Company

HAROLD S. KEMP
E. I. du Pont de Nemours & Company

EDWARD G. OPDYKE
Kay Fries Chemicals, Inc.

EXPERIMENTAL atmospheric boiling and condensing film coefficients are presented for water and normal hexane. Inside film coefficients for condensing steam also are given. The temperature variations around and along the boiling and condensing tubes are illustrated.

I. Introduction

A LABORATORY UNIT was constructed so that students could observe boiling and condensing and could experimentally determine boiling and condensing film coefficients. The data obtained during the first experimental program with the unit are presented in this paper. Atmospheric boiling and condensing film coefficients for water and normal hexane were determined. The temperature variations around and along the boiling and condensing tubes also were measured. Because of certain limitations of the apparatus the data are not precision measurements. However, since there appear to be no data in the literature concerning boiling film coefficients from a horizontal tube to normal hexane and concerning the temperature variations around a boiling tube, the results from the above-mentioned experimental program are presented for publication.

II. Experimental Work

A. *Materials Used*

The water used was distilled water. The normal hexane was technical-grade material obtained from Phillips Petroleum Company; its purity was given as 95 plus mole percent, the principal diluent being methylcyclopentane.

B. *Apparatus*

A diagrammatic flow sheet of the apparatus is presented in Figure 1.

1. *Still and Condenser.* The apparatus consisted essentially of a single-tube still and a single-tube condenser. The still was 21¼ inches long, and the condenser was 20⅝ inches in length. Both the still and condenser were constructed from standard 6-inch iron pipe, with standard 6-inch flanges and standard 6-inch steel end plates. The still and condenser each contained four windows which permitted visual observation of the boiling and condensing. The vapor-transfer line between the still and condenser was constructed from 2-inch iron pipe. A cap was placed over the vapor inlet in the condenser to distribute the vapor.

The tubes passed through the center of the still and condenser. They were copper tubes with a nominal diameter of 1.315 inches and an inside diameter of 0.951 inches. These are the dimensions of 1-inch extra-heavy iron pipe. Four copper-constantan thermocouples were spaced uniformly along each tube. The installation of these thermocouples was described and illustrated by Baker and Mueller.[1] The thermocouples were constructed from No. 30 copper and constantan wire, and the leads were protected by brass tubing which had a nominal diameter of 0.08 inches and an inside diameter of 0.05 inches. Collars, consisting of 4-inch lengths of 1½-inch iron pipe, were slipped over the tubes and the brass tubing containing the thermocouple leads. The annular space between each collar and tube was filled with solder. The distances between the collars on the still and condenser tubes were 21¼ and 20⅝ inches, respectively. Before the copper tube - thermocouple assemblies were installed, the tubes were cleaned with emory cloth. Packing glands at each end of the still and condenser made it possible to rotate the copper tube-thermocouple assemblies. Details of packing glands and collars were also described by Baker and Mueller.[1]

A copper trough was placed under the condensing tube to collect the condensate from the tube. A short trough and an umbrella were attached to each of the end plates in the condenser. The position of the short troughs was such that they collected the condensate from the extremities of the condensing tube and prevented this condensate from flowing down the faces of the end plates. The position of the umbrellas was such that condensate formed on the end plates was deflected away from the collecting troughs.

2. *Auxiliaries.* A preheater, preheating the cooling water, made it possible to vary the temperature of the cooling water and the temperature differential across the condensing film. An auxiliary condenser was provided so that the non-condensable gases could be swept from the principal condenser. The condensate streams from the condensing tube, the condenser shell and the auxiliary condenser were passed through coolers in order to reduce the losses by evaporation and to insure a more accurate measurement of the quantity of each stream. A mist extractor was placed in the steam line, and the steam supply to the still was considered to be saturated.

C. *Experimental Procedure*

Measurements of the rate of flow and temperature rise of the cooling water, tube wall temperatures, temperatures in the still and condenser, temperature of feed to still, quantities and temperatures of condensate from condensing tube, condenser shell and auxiliary condenser and quantity and pressure of steam to the still were made at 45 degree intervals

TABLE 1
Experimental Data

RUN	CONDENSATE LB./MM.		MEAN TEMPERATURE OF TUBE °F.		TEMPERATURE °F.		STEAM			COOLING WATER TEMPERATURE °F.		Temp. °F. Feed to Still
	Off Tube	Total	Still	Condenser	In Still	In Condenser	Lb./Min.	Psia	Lb./Min.	Inlet	Outlet	
WATER												
1	0.679	0.736	232.6	195.6	211.6	210.7	0.796	31.7	23.89	55.0	82.8	140.2
2	0.687	0.742	233.05	201.3	211.2	210.5	0.863	30.6	19.97	53.6	86.9	124.0
3	0.427	0.508	229.5	205.7	210.8	210.5	0.663	26.5	21.25	114.8	134.35	111.0
NORMAL HEXANE												
4	0.693	1.146	172.8	102.8	155.0	154.0	0.293	14.4	23.69	56.1	61.06	97.0
5a	0.881	1.800	179.0	106.8	155.2	155.0	0.390	14.4	23.02	60.25	64.97	110.5
5b	0.792	1.710	177.7	127.4	155.7	155.2	0.363	14.4	23.67	104.15	107.03	113.4
6a	0.824	1.400	176.4	106.2	155.7	155.0	0.299	14.4	22.83	57.35	62.62	103.5
6b	0.604	0.876	171.9	112.5	155.7	154.5	0.178	14.4	23.01	75.70	80.11	95.0

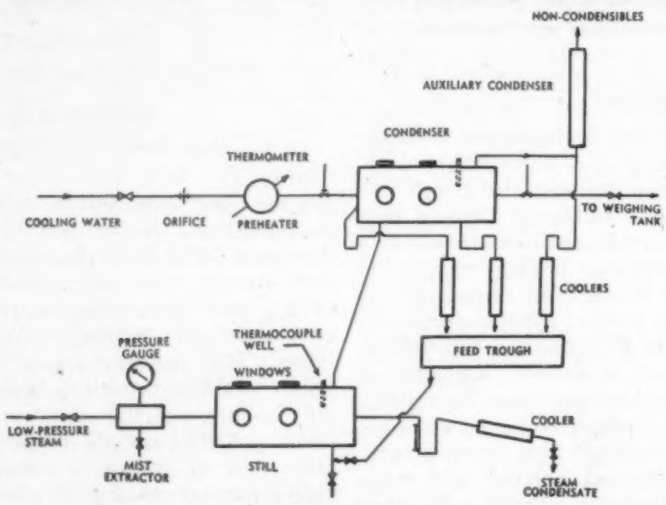

FIGURE 1
Diagrammatic flow sheet.

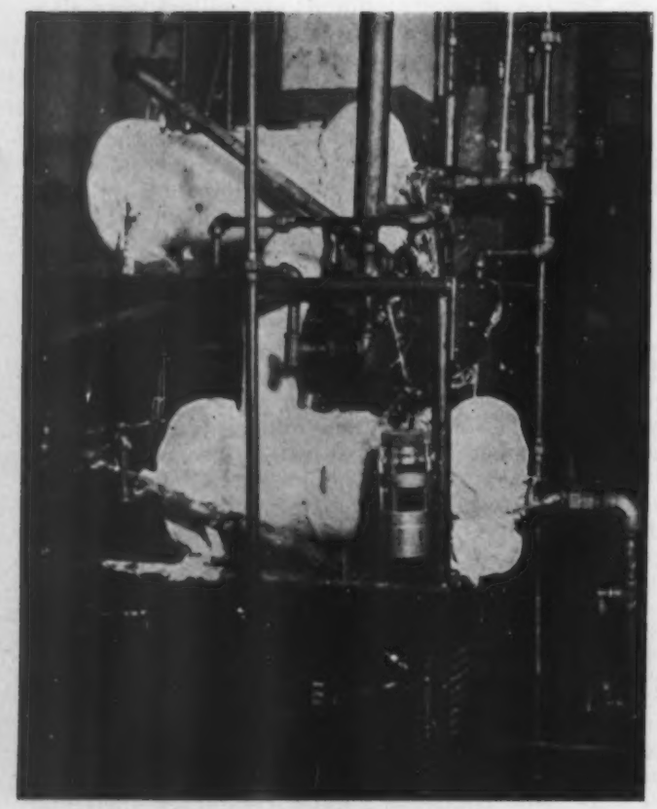

FIGURE 2-A

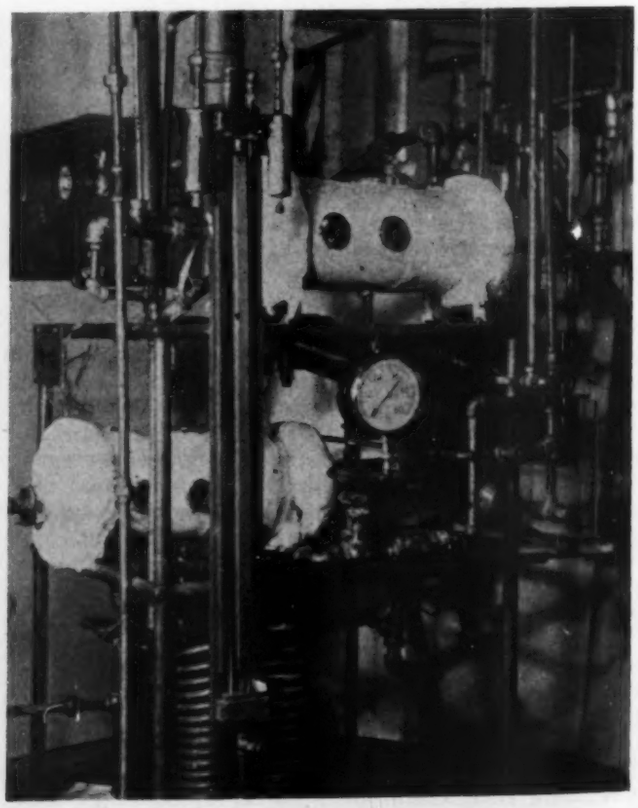

FIGURE 2-B

General views of apparatus.

TABLE 2

Boiling Film Coefficients for Water and Normal Hexane

RUN	HEAT TRANSFERRED Btu/Hr. Q_1	Q_2	TEMPERATURE DIFFERENTIAL °F. Δt_1	Δt_2	FILM COEFFICIENT Btu/(Hr.) (Sq. Ft.) (°F) h_1	h_2
WATER						
1.....	45,000	44,800	18.3	18.3	4030	4010
2.....	48,800	48,400	18.9	18.9	4230	4200
3.....	37,800	33,900	16.4	16.7	3770	3330
NORMAL HEXANE						
4.....	17,100	14,800	16.8	16.9	1660	1440
5a.....	22,700	22,900	22.4	22.4	1660	1680
5b.....	21,100	21,900	20.7	20.7	1670	1730
6a.....	17,400	15,500	19.6	19.8	1450	1280
6b.....	10,500	10,500	15.6	15.6	1100	1100

Note: Area of boiling film=0.610 sq. ft.

Values followed by subscript 1 were based on heat transfer as measured by the quantity of steam drips.

Values followed by subscript 2 were based on heat transfer as measured by the quantity of water or normal hexane vaporized and estimated heat loss in still.

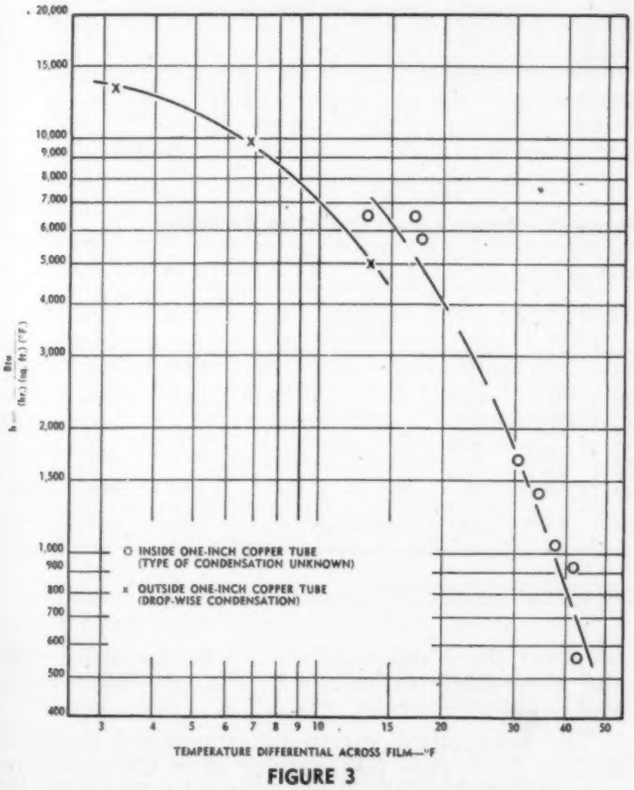

FIGURE 3

Coefficients of heat transfer—condensation of steam.

as the tubes were rotated. In connection with the study of normal hexane, rotation of the still tube was made at 90 degree intervals. Because of the activity in the still, rotation of the still tube at 455 degree intervals appeared superfluous.

The tube-wall thermocouples were calibrated stallation in order to detect any faulty thermocouples. They were calibrated again after the tubes and thermocouples were in place. Measurements were made with a semi-precision type potentiometer. The temperature rise of the cooling water was measured with thermometers which permitted readings to 0.1° C. The calibration of both thermocouples and thermometers was made using mercury thermometers which had been calibrated at the National Bureau of Standards. The pressure gage in the steam line was calibrated with a dead-weight tester.

The flow rate of the cooling water and the quantity of steam drips were determined by weighing. The

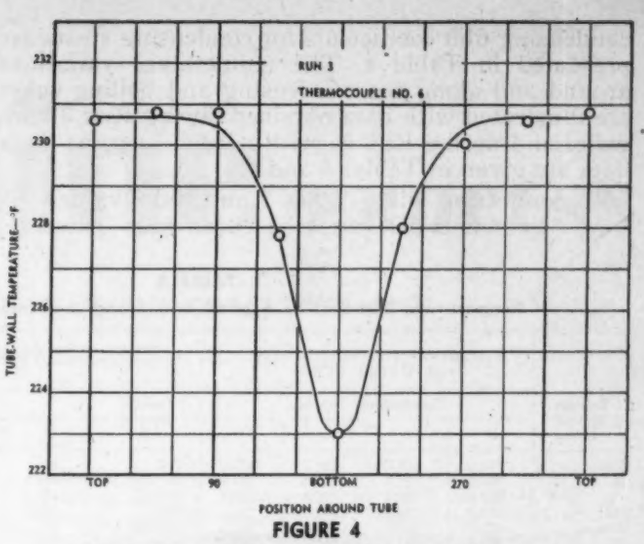

FIGURE 4

Temperature variations around tube when boiling water.

twice. A preliminary calibration was made before inquantities of condensate from the condensing tube, condenser shell and auxiliary condenser were determined from the volume and temperature of each stream.

III. Experimental Results

A summary of the experimental heat-transfer data is given in Table 1. Summaries of the outside boiling and condensing film coefficients for water and normal hexane are given in Tables 2 and 3. The inside

TABLE 3

Film Coefficients for Drop-Wise Condensation of Water and Film-Wise Condensation of Normal Hexane

RUN	HEAT TRANSFERRED Btu/Hr. Q_1	Q_2	TEMPERATURE DIFFERENTIAL °F. Δt_1	Δt_2	FILM COEFFICIENT Btu/(Hr.) (Sq. Ft.) (°F) h_1	h_2
WATER						
1.....	39,700	39,900	12.6	12.6	5,320	5,340
2.....	40,300	39,400	6.7	6.7	10,150	9,920
3.....	25,000	25,600	3.2	3.2	13,200	13,520
NORMAL HEXANE						
4.....	6,040	6,940	50.8	50.8	201	231
5a.....	7,680	6,520	47.7	47.8	272	230
5b.....	6,900	4,100	27.4	27.5	425	252
6a.....	7,250	7,230	48.3	48.3	254	253
6b.....	5,330	6,030	41.7	41.6	216	245

Note: Area of Condensing Film=0.592 sq. ft.

Values followed by subscript 1 were based on heat transfer as measured by quanttyi of water or normal hexane condensed on tube.

Values followed by subscript 2 were based on heat transfer as measured by heat absorbed by the cooling water.

TABLE 4

Inside Film Coefficient for Condensing Steam

RUN	HEAT TRANSFERRED Btu/Hr. Q_1	Q_2	TEMPERATURE DIFFERENTIAL °F. Δt_1	Δt_2	FILM COEFFICIENT Btu/(Hr.) (Sq. Ft.) (°F) h_1	h_2
WATER						
1.....	45,000	44,800	17.7	17.7	5760	5730
2.....	48,800	48,400	14.9	15.0	7430	7310
3.....	37,800	33,900	11.1	11.4	7700	6740
4.....	17,100	14,800	37.0	37.1	1050	910
5a.....	22,700	22,900	30.4	30.4	1690	1710
5b.....	21,100	21,900	31.8	31.7	1510	1570
6a.....	17,400	15,500	33.4	33.5	1180	1050
6b.....	10,500	10,500	38.4	38.4	620	620

Note: Area of condensing film=0.441 sq. ft.

Values followed by subscript 1 were based on heat transfer as measured by the quantity of steam drips.

Values followed by subscript 2 were based on heat transfer as measured by the quantity of water or normal hexane vaporized and estimated heat loss in still

condensing film coefficients for condensing steam are presented in Table 4. The temperature variations around and along the condensing and boiling tubes are illustrated with data obtained during Run 3 with water and during Run 6a with normal hexane; these data are given in Tables 5 and 6.

In connection with Tables 2 and 4 the values followed by subscript 1 were based on heat quantities as measured by the quantity of steam drips. The values followed by subscript 2 were based on the quantity of steam or normal hexane vaporized and the estimated heat loss from the still. In connection with Table 3 the values followed by subscript 1 were based on heat transfer as measured by the quantity of water or normal hexane condensed on the tube. The values followed by subscript 2 were based on the heat absorbed by the cooling water. Attention is directed to the fact that the inside and outside skin temperatures of the boiling and condensing tubes were calculated assuming the tube thermocouples were located exactly in the center of the tube wall.

The boiling film coefficients for water compare quite well with those published by Bonilla and Perry[2]. The boiling film coefficients for normal hexane appear to be the only data which have been published concerning this compound. In connection with condensing films attention is directed to the fact that drop-wise condensation was obtained with water and film-wise condensation was obtained with normal hexane. The film coefficients for drop-wise condensation of water compare quite well with those reported by Fitzpatrick, Baum and McAdams.[3] The experimental data concerning condensing film coefficients for normal hexane are correlated well with the Nusselt equation.[4]

Concerning the temperature variations around the condensing tube, the maximum tube-wall temperature was at the top of the tube, and the minimum temperature was at the bottom of the tube. The thickness of the condensate film was least at the top and greatest at the bottom of the tube. The average temperature along the condensing tube increased from the water-inlet end of the tube to the water-outlet end.

The temperature variations around and along the boiling tube were less regular than the variations around and along the condensing tube. In connection with water the maximum temperature was at the top of the tube and the minimum temperature at the bottom. The collection of condensate in the bottom of the tube contributed markedly to that variation of the tube-wall temperatures. The temperature along the boiling tube could be considered quite uniform. When vaporizing normal hexane with atmospheric steam, the temperature differential was large. There was much activity in the still, and this may explain the many temperature variations around the boiling tube. The average temperature at the steam inlet end of the tube was much higher than the average tube temperature at the other end. This indicated the possibility of film boiling at the steam-inlet end of the tube.

TABLE 5

Temperature Distribution Around and Along the Condensing Tube[a]

Position Around Tube	DROP-WISE CONDENSATION OF WATER (RUN 3) Thermocouple Number				Position Around Tube	FILM-WISE CONDENSATION OF NORMAL HEXANE (RUN 6a) Thermocouple Number			
	1[b]	2	3	4		1[b]	2	3	4
Top	205.0	203.7	208.2	208.6	Top	106.7	108.3	(c)	108.8
45	204.2	203.5	207.0	209.0	45	105.5	108.0	.....	108.8
90	204.6	203.1	207.3	209.0	90	104.2	106.8	.....	107.5
135	204.1	202.8	207.5	208.3	135	103.0	105.4	.....	106.1
Bottom	202.7	201.8	206.8	207.4	Bottom	101.8	103.6	.....	104.6
225	205.1	201.7	207.3	208.0	225	103.0	104.1	.....	105.8
270	204.1	201.8	207.6	208.0	270	104.7	105.5	.....	107.2
315	205.7	203.7	207.6	208.5	315	105.5	106.0	.....	108.5
Top	204.5	202.7	208.0	207.9	Top	106.3	108.0	.....	108.8
Average...	204.4	202.8	207.5	208.3	Average...	104.5	106.2	.....	107.4

[a] All temperatures are given in degree F.
[b] Thermocouple Number 1 was located nearest the inlet of the cooling water.
[c] Thermocouple Number 3 became shorted during investigation of normal hexane.

TABLE 6

Temperature Distribution Around and Along the Boiling

Position Around Tube	WATER (RUN 3) Thermocouple Number				Position Around Tube	NORMAL HEXANE (RUN 5[b]) Thermocouple Number			
	5	6	7[c]	8[b]		5	6	7	8
Top	232.0	230.6	223.9	232.5	Top	161.5	169.2	187.7	193.5
45	231.5	230.8	223.5	229.7	.....	.....	.....	.....	.....
90	231.5	230.8	223.5	229.2	90	161.8	176.6	186.8	192.8
135	227.9	227.8	222.8	229.1	.....	.....	.....	.....	.....
Bottom	224.8	223.0	221.2	224.7	Bottom	164.1	169.5	176.6	177.0
225	228.9	228.0	222.8	225.3	.....	.....	.....	.....	.....
270	231.2	230.1	223.5	226.7	270	162.2	179.4	187.1	160.6
315	231.2	230.6	223.3	229.0	.....	.....	.....	.....	.....
Top	231.5	230.8	223.6	231.8	Top	161.5	166.7	188.3	194.6
Average...	230.5	229.2	223.1[c]	228.7	Average...	162.2	172.3	185.3	187.

[a] All temperatures are given in degrees F.
[b] Thermocouple Number 8 was located nearest the steam entrance.
[c] During the investigation with water thermocouple 7 was not considered reliable.

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THE Synthine PROCESS

II. THE CATALYTIC SYNTHESIS, INCLUDING CATALYSTS AND REACTION MECHANISMS

JOHN C. LANE
Gulf Research & Development Company
and
B. H. WEIL
Georgia School of Technology

THIS is the second in a series of four articles on the Synthine process being prepared by B. H. Weil and John C. Lane. The authors are, respectively, Chief of the Technical Information Division, State Engineering Experiment Station, Georgia School of Technology, and Head of the Information Section, Chemistry Division, Gulf Research & Development Company.

THE first article in this series has dealt with the history of the Synthine process and with the various methods and raw materials employed in the preparation of synthesis gas. Once a suitable gas has been prepared, regardless of its source, the next step in the Synthine process is the catalytic synthesis, which consists of the hydrogenation of carbon monoxide over such catalysts and under such conditions that the reaction product consists of hydrocarbons of various chain lengths and degrees of saturation. Depending upon a number of factors, chiefly the ratio of hydrogen to carbon monoxide in the synthesis gas and the type of catalyst employed, one or the other of the following reactions predominates in the synthesis (although both almost always take place to some extent):[147]

$$nCO + (2n + 1)\,H_2 \rightarrow C_nH_{2n+2} + nH_2O \quad (1)$$

$$nCO + 2nH_2 \rightarrow C_nH_{2n} + nH_2O \quad (2)$$

The first of these reactions is favored by relatively high ratios of hydrogen to carbon monoxide and catalysts of strong hydrogenating power, while the second is favored by low hydrogen-carbon monoxide ratios and catalysts of less strong hydrogenating ability.

When iron catalysts are used instead of those containing nickel or cobalt, the reaction becomes:[193]

$$2nCO + nH_2 \rightarrow C_nH_{2n} + nCO_2 \quad (3)$$

As might be expected, since the Synthine reaction is exothermic and involves a reduction in volume, relatively low temperatures and somewhat elevated pressures favor the reaction. The synthesis is generally conducted at temperatures of approximately 375° F. with nickel- or cobalt-containing catalysts and at about 465° F. with iron catalysts. Except in cases where a specific product is desired (such as high-molecular-weight wax), the usual operating pressure lies somewhere between zero and 150 psig. The synthesis gas is passed through reaction chambers in one or more stages, with or without recycle of the unconverted portion;[26] the heavier products are condensed from the residual gas; and the lighter products are adsorbed on charcoal (in the atmospheric synthesis) or absorbed in oil (in the medium-pressure synthesis). Considerable variations in the quantity and types of products are obtained under different reaction conditions, as will be brought out in subsequent sections of this article. However, prior to discussion of the effect of the reaction variables, it might prove instructive to review the theories advanced on the mechanism of the process and to mention the various types of catalysts which may be employed.

Reaction Mechanisms

Most of the theories regarding the reaction mechanisms of the Synthine process stipulate the intermediate formation and decomposition of carbides of the catalyst metals. It has been shown that iron, cobalt, nickel, and ruthenium (those metals which best catalyze the reaction) all possess the common property of forming relatively unstable carbides of nonionic crystal structure when exposed to carbon monoxide at temperatures within the range employed in the Synthine process.[209] Below about 660° F., these carbides will react with hydrogen, being quantitatively converted to methane plus a small amount of ethane.[183, 184, 185] However, at temperatures above 660° F., the carbides tend to decompose to carbon.[133] In view of this behavior of the carbides of Synthine-catalyst metals, it may be interesting to note that the optimum temperature ranges for the synthesis reaction lie below 660° F., the temperature above which the carbides break down to carbon.[133]

Fischer[195] has long been of the opinion that methylene radicals, $(CH_2)_n$, are formed when the carbides are treated with hydrogen and that the products of the synthesis are formed by the combination of these radicals into chains of various lengths and degrees of saturation. At least as late as 1935, he was not willing to choose between two possible explanations for the mechanism of methylene-radical formation from the catalyst carbides.[195] He assumed either: (1) that the normal carbide, Me_3C, is formed in the presence of carbon monoxide and then reduced by hydrogen to the metal, giving rise to methylene radicals, or (2) that the normal carbide, Me_3C, is carbonized to a higher carbide, Me_3C_2, which, upon reduction to the normal carbide, forms the (CH_2) radicals. According to Fisher's beliefs, this step of the reaction

with nickel or cobalt catalysts may be represented by the equation:[193]

$$CO + 2H_2 \rightarrow (CH_2) + H_2O\ (+ 175\ Btu)$$

and, with iron catalyst, by the equation:

$$2CO + H_2 \rightarrow (CH_2) + CO_2\ (+ 173\ Btu)$$

Fischer's reasoning may be summed up as follows: since only Group 8 metals which are known to react with carbon monoxide at temperatures near 390° F. are suitable as Synthine catalysts, and since the carbides of these metals are found in the spent catalysts, it appears that carbides are formed by the interaction of the catalytic metals with carbon monoxide and that the methylene raidcals are produced by secondary hydrogenation of these carbides.[191, 192] In 1938, Fischer reiterated his assumption that the mechanism may involve the alternate formation of higher and lower carbides.[193]

As mentioned in the introduction to this series, one of the current theories presupposes that the catalyst, in addition to giving rise to methylene radicals, also influences both their polymerization to hydrocarbons and the subsequent hydrogenation of these hydrocarbons.[140] Experiments reported by Herington and Woodward[210] indicate that two types of active centers are present on the surface of a cobalt Synthine catalyst. The Type A center (probably cobalt carbide) is considered responsible for the formation of methylene radicals and their polymerization to hydrocarbons. The Type B center (probably metallic cobalt) is believed to provide the surface upon which the hydrogenation and chain-liberation occurs. "The predominance of one or the other type of center explains some of the problems of preliminary methane formation and catalyst poisoning."[140]

Komarewsky and Riesz[90] are also of the opinion that Synthine catalysts must possess both a polymerizing and a hydrogenating effect, in addition to the ability to form carbides of suitable stability. They have pointed out that nickel, cobalt, and iron form suitable carbides and possess hydrogenating ability; when pure nickel is used as catalyst, however, only methane is formed in the synthesis step, and hydropolymerization to form higher hydrocarbons does not take place. In such cases, a multi-component, "complex-action" catalyst seems required, and this is borne out by the fact that the catalyst compositions most favored for the Synthine reaction are, as mentioned, composed of several elements.

Experiments reported by Craxford and Rideal have seemed to indicate that the reaction between carbon monoxide and cobalt to form cobalt carbide is far too slow to explain the rate of synthesis of hydrocarbons from hydrogen-carbon monoxide mixtures.[187, 188] However, recent tests conducted by the United States Bureau of Mines indicate that the rate of carbiding is of the same order of magnitude as the synthesis rate.[237] Craxford's suggested mechanism for the cobalt-catalyzed synthesis assumes that the first step is the reaction of both carbon monoxide and hydrogen to form the carbide:[187, 188]

$$2Co + CO + H_2 \rightarrow Co_2C + H_2O$$

This reaction (which occurs more rapidly than carbiding with carbon monoxide alone) involves: (1) chemisorption of carbon monoxide on the catalyst, (2) reduction of the chemisorbed carbon monoxide by hydrogen to give the carbide, and (3) reduction of the carbide by molecular hydrogen to chemisorbed methylene. In all their experiments, Craxford and Rideal noted a parallel relationship between ortho-para hydrogen conversion and methane formation. When methane was the main product, the ortho-para conversion was high (an indication of the presence of chemisorbed hydrogen); conversely, when higher hydrocarbons were being produced, the ortho-para conversion amounted to only a few percent. This led them to the belief that a relationship may exist between the quantity of chemisorbed hydrogen on the catalyst surface and the products of the synthesis; i.e., that, in the presence of a large amount of chemisorbed hydrogen, methane is formed from the reaction; while in the presence of smaller quantities, reduction of the carbide (presumably by molecular hydrogen) occurs with the production of higher hydrocarbons. The latter step may occur through the formation of polymethylene macro-molecules on the catalyst surface, their reduction by molecular hydrogen, and subsequent desorption.

Storch[133] has pointed out the possibility that hydrogen may catalyze the formation of metal carbides by forming an unstable metal hydride. "This may result in sufficient distortion of the metal lattice so that, upon removal of the hydride hydrogen (by decomposition or by reaction with neighboring metal carbide or adsorbed carbon monoxide), penetration of the lattice by carbon monoxide is accelerated." The fact that iron catalysts sintered in hydrogen at 1560° F. are quite active is pointed out by Storch[237] as lending weight to the premise that only a very small fraction of the catalyst surface possesses the critical spacing of metal atoms which is necessary to facilitate the synthesis reaction.

Summing up what is known or assumed of the reaction mechanism of the Synthine process, and attempting to explain the synthesis reaction in the light of the carbide-methylene radical theory, carbide formation would seem to be the first step, followed by establishment on the catalyst surface of a "steady state" which involves polymerization, reduction, and desorption of the methylene and polymethylene groups, the extent of the polymerization being determined by the rate of desorption. Low conversion of the catalyst metal to its carbide and/or a rapid rate of desorption of the methylene groups cause a low concentration of methylene groups on the catalyst surface, and polymerization is consequently retarded. Under these conditions, methane is the main product, and ample active surface is provided on the catalyst surface for ortho-para hydrogen conversion. The high production of methane (to the exclusion of higher hydrocarbons) which is experienced when breaking in a new catalyst is explained by the necessity for covering the catalyst surface with polymethylene groups to slow up the reduction of the methylene radical to methane. The accelerated rate of desorption of methylene groups at higher temperatures might account for the fact that methane production predominates at temperatures above 480° F.[132]

The reaction mechanism proposed by Matsumura, Tarama, and Kodama[221] assumes the transient existence of the metal oxide in the catalyst-carbiding step and the subsequent formation of chemisorbed water by the reduction of this oxide with hydrogen from the synthesis gas. These investigators consider the first step of the reaction to be:

$$CO + 3Me \rightarrow Me_2C + MeO$$

Their explanation for the formation of hydrocarbons is not markedly different from those of the catalyst-

carbide theories previously discussed. However, the interesting feature in their theory is the explanation it affords for the formation of water as a reaction product of the synthesis over cobalt, and of carbon dioxide from the reaction over iron. They believe that:

(1) Through combination with atomic hydrogen adsorbed on the catalyst, the carbide forms methylene groups which, during their residence upon the catalyst, are polymerized, reduced, and converted into hydrocarbons. The hydrocarbons so formed, present in the adsorbed state on the catalyst, eventually become desorbed. The three processes (polymerization, reduction, and desorption) occur successively in respective order, attaining a sort of "steady state" in which hydrocarbons of various molecular weights are produced.

(2) Hydrocarbons of the gasoline range are formed over cobalt catalysts at temperatures above 320° F. because only above this temperature are sufficient quantities of active hydrogen adsorbed on the catalyst.

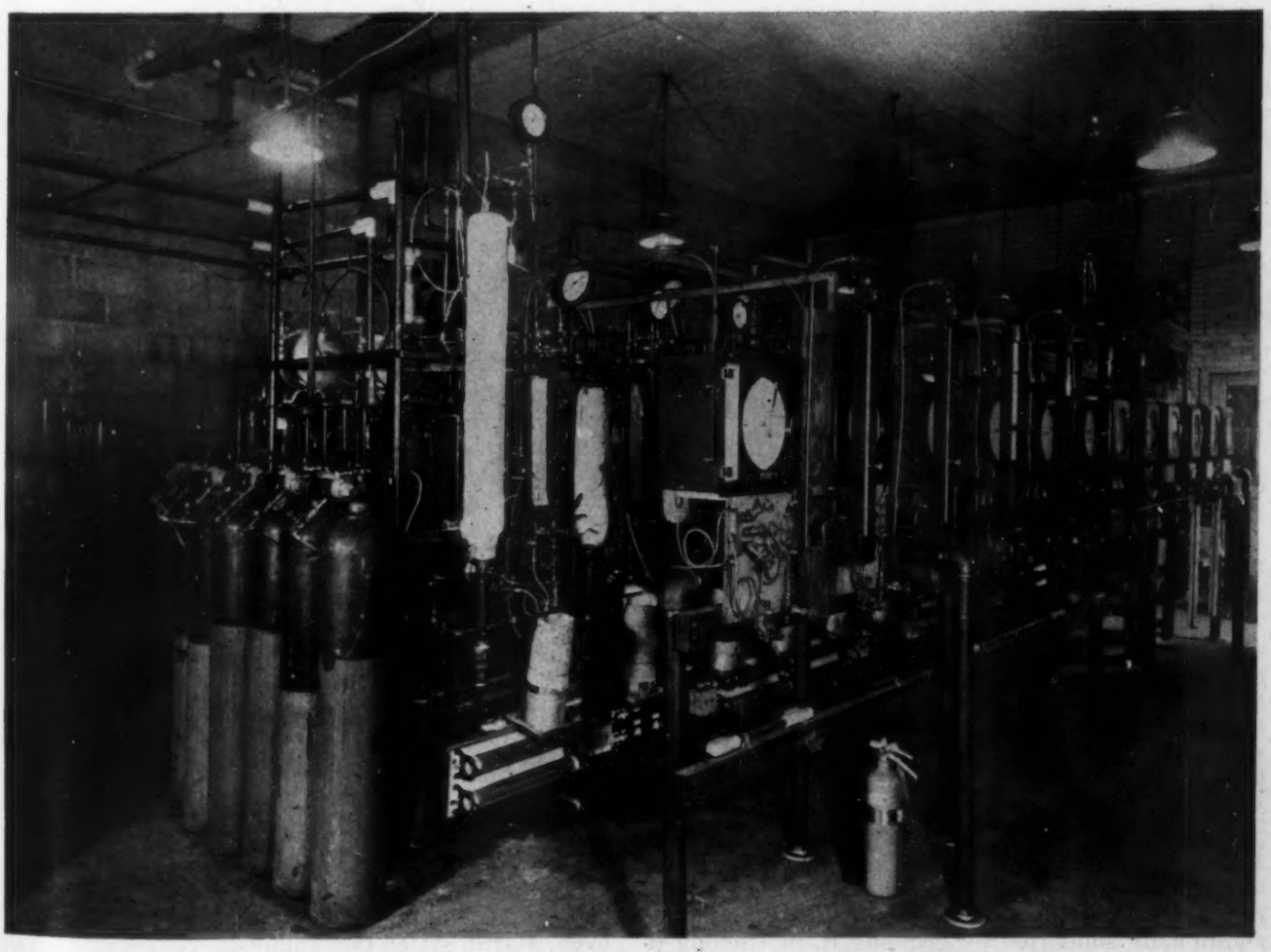
(3) The higher reaction temperature required when iron catalysts are used may be explained by the fact that the temperature of formation of iron carbide from the metal and carbon monoxide is greater than that for cobalt carbide formation.

(4) The subsequent reduction with hydrogen of the metal oxide formed in the catalyst-carbiding step gives rise to water which is chemisorbed on the catalyst surface. With cobalt catalysts, the rate of desorption of chemisorbed water is greater than with iron catalysts. Thus, in the case of the reaction over cobalt, the chemisorbed water is rapidly desorbed and appears as a reaction product. However, when iron catalysts are used, there is greater opportunity for the carbon monoxide in the synthesis gas to form carbon dioxide by reaction with the metal-oxide areas on the catalyst surface, accounting for the appearance of carbon dioxide in the reaction products.

Catalysts

The catalytic metals used by Fischer and Tropsch in their early experiments were iron and cobalt, the former being activated with copper and alkali and the latter with zinc oxide. Their experience using nickel was disappointing, and they attributed this fact to the known powerful hydrogenating influence of nickel, believing that it caused direct hydrogenation of the carbon monoxide to methane. However, they later found nickel to yield liquid hydrocarbons when combined with other catalytic materials.[195] In the light of the theory that a Synthine catalyst must encourage both polymerization and hydrogenation, the failure of nickel alone to catalyze the synthesis of higher hydrocarbons might be explained, as mentioned, by its lack of polymerizing ability.

While admixture with other catalytic or activating materials is necessary in the case of nickel, it is also highly desirable in the case of the other catalytic metals. The added materials may serve one or more of the following purposes; they may: (1) supply a catalytic effect not possessed by the catalytic metal alone; (2) activate or promote the catalyst; (3) facilitate catalyst preparation, conditioning, or regeneration; (4) inhibit catalyst poisoning; (5) improve the physical nature of the catalyst; and/or (6) provide a support for the catalytic metal. Superior results have been obtained with those multicomponent catalysts in which (1) each component has been selected for its particular effect upon the reaction and (2) the relative proportions of these components have



Courtesy H. H. Storch, U. S. Bureau of Mines

Catalyst testing units in Bureau of Mines Synthine laboratory, showing recording and indicating flowmeters.

been adjusted to give composite masses capable of directing the synthesis toward the production of hydrocarbons of desired molecular weight and structure. The practical application of the Synthine process already requires "complex-action" catalysts,[216] and it would logically appear that the development of modifications of the process to yield special-type hydrocarbons will, in the future as in the past, largely depend upon the results of catalyst research and development.

The choice of the catalytic metal has, in itself, a definite influence on the degree of saturation of the products of the synthesis. Cobalt is intermediate in hydrogenating power, its replacement by nickel in Synthine catalysts leading to the formation of more saturated products and its replacement by iron resulting in greater yields of olefins.[195]

The theoretical yield from one cubic meter of "ideal" synthesis gas ($2H_2 + 1CO$) is 208 grams (about 13 pounds per 1000 cubic feet). However, in actual practice, the synthesis gas always contains a considerable quantity of inert materials (CO_2, N_2, CH_4, etc.); for this reason, even with a catalyst giving total conversion, the yield will rarely exceed about 180 grams (11.2 pounds per 1000 cubic feet).[123]

The more active Synthine catalysts consist of mixtures of cobalt and/or nickel with activating materials such as alumina, thoria, or other difficultly reducible metal oxides. These catalysts give fairly high yields (up to about 170 grams per cubic meter) at temperatures of about 374° F. Recent experiments with iron catalysts have shown that at higher temperatures and pressures, such as 465° F. and 150 pounds per square inch, yields may be obtained which are comparable in quantity to those from cobalt or nickel complex catalysts.[129]

Supported Nickel Complex Catalysts

Complex catalysts containing nickel or cobalt as the prime catalytic metal and other elements and compounds as supplemental catalytic agents are generally prepared by coprecipitation of the chemically active ingredients upon the inert carrier. One such method involves the addition of alkali carbonates to solutions of the nitrates of the desired components in the presence of sufficient kieselguhr to serve as a support. During the precipitation, the kieselguhr is kept in suspension by mechanical agitation; the mixture is then filtered, washed, dried, and reduced with hydrogen. In the case of nickel-manganese-alumina catalysts, carrying out the preciptation in the presence of ammonia is said to increase the yield in the subsequent synthesis step and to lower the temperature for optimum reduction of the catalyst from about 840° F. to 570-660° F.[199]

Catalysts consisting of 100 parts nickel; 20 parts manganese oxide; 4-8 parts thoria, alumina, tungstic oxide, or uranium oxide; and 125 parts kieselguhr have been found to yield from 0.75-1.2 gallons of liquid hydrocarbons per 1000 cubic feet of synthesis gas at temperatures of 365-410° F. and a space velocity of about 150 volumes of synthesis gas per volume of catalyst per hour.[205, 212, 252]

In its search for a longer-lived catalyst, Robinson-Bindley Processes, Ltd. (now known as Synthetic Oils, Ltd.) developed a nickel-manganese-alumina catalyst precipitated on kieselguhr. This catalyst was additionally supported by grinding it, moistening it with a partly hydrolyzed solution of ethyl orthosilicate in toluene, and extruding it in rods through a quarter-inch nozzle. Upon exposure to moist air for several days, the hydrolysis of the ester was complete, and a porous silica-kieselguhr-supported catalyst was obtained.[179, 186]

Fischer early patented complex nickel catalysts prepared by the coprecipitation technique, specifying thorium, aluminum, and cerium compounds as suitable activating materials.[256, 273] Russian investigators have also studied complex nickel catalysts.[213, 230]

Supported Cobalt Complex Catalysts

Kieselguhr-supported mixtures of cobalt and thorium oxide (and/or other activating materials) are the "standard" catalysts for the Synthine process. Such catalysts give high yields of primary product and, in their present state of development, possess fairly long active lives. The catalyst used exclusively by the Germans in their commercial-scale Synthine plants during the late war was of this type, and a description of its composition, preparation, conditioning, regeneration, and reworking may be found in the section of this article dealing with captured German information. According to one interrogated German, "It has been possible in a modern synthesis plant (using the wartime catalyst) to produce 168 grams of primary product per cubic meter of synthesis gas, and also to obtain approximately 94 percent of the theoretical yield of 180 grams (from synthesis gas containing inerts)."[147] Expressed in English units, this is a yield of 10.5 pounds per 1000 cubic feet out of a possible 11.2 pounds.

In 1935, after testing "several hundred" catalysts, Fischer[195] stated that cobalt-thorium-kieselguhr catalysts had given the best results up to that time. He also reported that the addition of two percent of copper to the catalyst greatly increased the ease of reduction of the catalyst. The experiments of Fischer and his co-workers with the Co-Cu-ThO_2-kieselguhr catalyst have been reported by both Koch and Pichler.[214, 226]

Several Japanese investigators have studied kieselguhr-supported cobalt catalysts, reporting quite high yields from compositions consisting of 5-10 percent of copper, 4-12 percent of manganese oxide, and 4-12 percent of thoria, alumina, or uranium oxide.[205, 212, 252]

As mentioned, all commercial-scale Synthine operations in Germany appear to have been conducted over a more or less standard type of cobalt-thoria-magnesia-kieselguhr catalyst.[26] Upon questioning, a number of German technical men maintained that all synthesis plants in Western Germany were supplied with a catalyst cotaining 100 parts Co, 5 parts ThO_2, 8 parts MgO, and 180-220 parts kieselguhr.[26, 27] However, a tentative analysis of what was said to be fresh catalyst obtained from Ruhrchemie at Oberhausen-Holten indicated 8.8 parts ThO_2, 4.4 parts MgO, and 260-330 parts kieselguhr to every 100 parts of Co.[27]

Magnesia-containing catalysts are said to have been in use since 1938, their introduction having made possible catalyst lives of up to eight months at normal pressure.[27] According to Dr. F. Martin, managing director of Ruhrchemie A.-G., the magnesia was added solely to increase the hardness of the resulting catalyst and thus to reduce its tendency to disintegrate to dust in the ovens, since such dust causes bad gas distribution and leads to "hot spots" in the oven. Martin stated that catalysts containing the full amount of thoria produced more paraffin and that

Courtesy W. C. Schroeder, U. S. Bureau of Mines

Synthine catalyst manufacturing plant at Sterkrade-Holten, the only commercial catalyst plant in Germany.

the substitution of magnesia decreased this production, it being necessary to strike a compromise between the relative proportions of thoria and magnesia in order to obtain a catalyst giving high production and possessing good physical strength. Prof. Dr. Alberts, director at Castrop-Rauxal and former manager of the Sterkrade-Holten plant, said that the incorporation of magnesia in the catalyst also contributed to the east of initiating the synthesis and to the depression of methane and carbon formation.[26]

The kieselguhr used in such catalysts reportedly must contain less than 1 percent of iron or excessive methane production will be obtained during the synthesis. Its content of uncalcined aluminum trioxide also must be low (0.4 percent or less), otherwise the catalyst tends to gel. German practice was to calcine the kieselguhr, prior to use, at 1100-1300° F., after which it should not contain over 1 percent total volatile matter, including water. Although the iron in the kieselguhr may be removed by acid treatment, it was believed that the advantage gained by the iron removal is overbalanced by the loss of desirable physical structure when acid treatment is employed.[26]

The catalyst was prepared from a solution of the corresponding nitrates (in suitable proportion) by precipitation at about 212° F. with sodium carbonate. After washing, drying, and screening, the finished catalyst particles were 1-3 millimeters (0.04-0.12 inch) in size and had a bulk density of 320-350 grams per liter (20-21.8 pounds per cubic foot). Reduction of the catalyst was reportedly effected by treating it with ammonia synthesis gas (H_2, 75 percent; N_2, 25 percent) preheated to about 860° F. at a space velocity of around 8800 for some 40-60 minutes. Optimum regeneration conditions were said to be difficult to determine; in general, the lower the reduction temperature the better, but lower temperature require the use of longer times.[26] A similar process of catalyst manufacture (carried out at the Carrieres-Kuhlman plant at Harnes, France) has been described in detail.[123]

According to the information obtained, the spent catalyst from all Western Germany's synthesis plants was returned for reworking to Ruhrchemie at Sterkrade-Holten, the plant where it was produced. A flow diagram of this catalyst-reworking plant has been published.[26] The components of the spent catalyst were converted to the corresponding nitrates by reaction with nitric acid (about 36° Baume), then treated to recover the cobalt and thorium. The magnesium was discarded, since its value was not considered great enough to warrant the effort of purification. Details of the catalyst-recovery operation have been outlined by Aldrich.[123, 180]

Supported Cobalt-Nickel Complex Catalysts

As previously mentioned, cobalt catalysts give lower methane yields and more olefins in the product than do nickel catalysts. However, about 50 percent of the cobalt may be replaced by nickel without loss of these advantages.[247] In his experiments with precipitated catalysts containing equal parts of cobalt and nickel as the prime catalytic agents, Tsutsumi [250, 251, 253, 254] obtained very low yields without the use of promoters. His most satisfactory catalyst contained 20 percent manganese oxide, 20 percent uranium oxide, and 125 percent kieselguhr (based on the total weight of cobalt plus nickel). The activity of a cobalt-nickel catalyst promoted with the oxides of manganese, uranium, and thorium was found to be increased approximately 10 percent by a slow oxidation at 70° F prior to its reduction.[211]

Courtesy W. C. Schroeder, U. S. Bureau of Mines

Hoesch-Benzine contact oven.

"Alloy-Skeleton" Catalysts

The heat-transfer problem involved in carrying out the highly exothermic Synthine process over the poorly conductive, "conventionally" supported catalysts has prompted considerable investigation of catalysts which, by virtue of their finely divided state or their physical structure, permit a more rapid distribution of the heat liberated during the reaction. Highly porous, catalytically active "skeleton" catalysts consisting of nickel or cobalt or alloys of both and possessing a greater coefficient of heat transfer than precipitated catalysts have been developed by Raney.[229, 276, 277, 279] These catalysts are prepared by alloying nickel and cobalt (singly or mixed) with aluminum or silicon and subsequently removing the latter material by solution in aqueous caustic. Experiments with cobalt-nickel skeleton catalysts have indicated that (1) the optimum ratio of cobalt to nickel is 1:1, (2) catalysts prepared by alloying with silicon possess higher activity than those in whose preparation aluminum is used, and (3) the presence of even small amounts of copper or manganese in the catalyst is undesirable.[200] It has been shown that skeleton catalysts prepared from alloys of nickel with aluminum, nickel with silicon, nickel with manganese and silicon, and nickel with iron and aluminum are decidedly inferior to those prepared from nickel-cobalt-silicon alloys. The crystal structure of the catalyst obtained from a nickel-cobalt-magnesium alloy, upon microscopic investigation, has been found to be much coarser than that of the catalyst prepared from the corresponding silicon alloy.[240] The activity of skeleton catalysts prepared from nickel-aluminum, cobalt-aluminum, and cobalt-silicon alloys has been investigated by Rapoport and Polozhintseva.[231] Detailed studies of skeleton catalysts have been made by Japanese investigators[224, 225, 238, 240, 242, 243, 244, 245, 246], who found that the yield of liquid hydrocarbons with such catalysts amounts to about 5.8 pounds per 1000 cubic feet of synthesis gas.

The preparation of catalyst pellets having an "integral, porous, metallic skeleton" has been described in U. S. 2,136,509.[280] The method involved is somewhat different from that devised by Raney. The finely divided oxide of a catalytic metal (e.g., nickel or iron oxide) is mixed with a finely divided metal (e.g., iron or copper), the mixture is compressed into pellets, and the pellets are heated in a reducing atmosphere at such temperature and for such time as is required to reduce most of the metal oxide to the catalytic metal and to cause alloying of the two metals, "the temperature and time of heating being insufficient to cause all of the catalytic metal of the oxide to alloy with the other metal and the temperature being insufficient to cause sintering of the other metal in the absence of the catalytic metal."

Catalysts of the "alloy-skeleton" type deteriorate more rapidly and yield (at best) about 20 percent less liquid product than precipitated catalysts having the same catalytic ingredients.[205, 212, 252] However, as mentioned, their ability to conduct heat more rapidly than precipitated catalysts has stimulated interest in their possibilities for the Synthine process.

Suspended Catalysts

The transfer of heat from the catalyst surface also may be facilitated by the use of finely divided catalyst particles suspended in a liquid medium. When the temperature of any given catalyst particle rises to a sufficient level, the excess heat from the reaction, instead of overheating the catalyst, is converted into heat of vaporization of the liquid. A catalyst consisting of iron, magnesium oxide, and zinc oxide suspended in anthracene oil is claimed to yield hydrocarbons heavier than gasoline (i.e., lubricating oil and paraffin wax) at temperatures below 700° F. and pressures above 300 psi.[260] The use of a nickel-aluminum-kieselguhr catalyst suspended in a heavy, sulfur-free oil is said to result mainly in the production of methane.[274]

In one apparatus designed for the use of suspended catalysts, the catalyst is arranged in layers in a vertical tube, and a reflux condenser is provided at the

upper end of the tube to return condensed fractions of the reaction product to the reaction zone, a substantial portion of this reflux consisting of oils which vaporize under the prevailing reaction conditions. The liquid medium is maintained at a level above the top layer of the catalyst, and the synthesis gas is introduced at the lower end of the tube. The catalyst prepared by fusing a mixture of iron powder, silicon powder, titanium oxide, and potassium permanganate in a stream of oxygen (thereby converting the Fe to Fe_3O_4), cooling and comminuting the resulting mass, and treating the resulting particles with hydrogen is said to be suitable for the production of hydrocarbon oils boiling in the range of 105-570° F at reaction conditions of 680-715° F. and 1470 psi.[261, 281] Another technique involves introduction of the liquid medium into different zones of the reaction space.[269] The liquid medium may first be passed along the catalyst chamber "in indirect heat-exchange relation with the reaction participants and then through the chamber in the same direction as the reaction participants. Preferably, the liquid medium may be passed along the reaction chamber in countercurrent flow to the reaction participants so that, while the liquid medium is at its lowest temperature, it comes into contact with the hottest part of the catalyst chamber, thereby keeping the temperature of the chamber much more uniform."[270] The liquid-phase catalytic synthesis has been discussed by Fischer and Pichler.[203]

There are two main disadvantages of conducting the reaction in a liquid medium: (1) the difficulty of removing the reaction products and (2) the larger space requirements. Where the liquid medium is, itself, a fraction of the reaction product, the problem is less difficult than in the case where some other liquid is employed. Distillation or solvent extraction has been suggested for the latter case. Where the liquid medium differs markedly in chemical structure from the synthesis products; e.g., the medium is aromatic and products are paraffinic, highly selective solvents (such as sulfur dioxide) may be used.[260] Standard Oil Development Company[265] has patented a process, designed to facilitate removal of the reaction products, in which the reaction is carried out in a 50-tray, bubble-cap tower with a catalyst suspension consisting of about three quarters of a pound of finely divided nickel catalyst activated with magnesium and aluminum oxides per gallon of sulfur-free paraffin wax. The yield is said to amount to about one gallon of liquid hydrocarbons per 1000 cubic feet of synthesis gas charged.

"Fluid" Catalysts

Perhaps the most recent development in the quest for a Synthine catalyst possessing superior heat-transfer characteristics is the "fluidized" catalyst, which consists of very finely divided particles suspended in the synthesis-gas stream. The experience gained in operation of the many (over 32) commercial-sized Fluid Catalyst Cracking units[217, 235] already in operation should provide much of the "know how" for the analogous Synthine process.

Using such a catalyst for the Synthine process, it is claimed that the exothermic heat of reaction may be removed "immediately as it is formed along the path of the flowing gases in the reaction zone through the walls of the reaction zone by circulating a cooling medium without the walls of the reaction zone."[285]

In U. S. 2,347,682,[287] which specifies this type of catalyst, it is said that the temperature can be kept substantially constant anywhere within the range of 225-425° F. by directly admixing relatively cool materials with the mixture in the synthesis zone. Unreacted gases and low-molecular-weight hydrocarbon gases are separated from the catalyst and recycled. Cool catalyst may be used to control the temperature of the reaction zone. A very recent patent[289] specifies the circulation of a solid heat carrier through the reactor, along with the fluidized catalyst. The heat carrier must have a higher separating rate than the catalyst, in order to permit separation, cooling, and recirculation of the solid heat carrier.

Standard Catalytic Company's "Chemican Process,"[288] a fluid-catalyst-type Synthine operation, maintains a pressure head at the bottom of the column containing the powdered catalyst by injecting an inert gas at one or more points. The pressure at the bottom of the column is said to be a direct function of the density of the catalyst powder and the height of the column. "Using 200-400 mesh activated clay having deposited thereon iron oxides, the pressure obtained was one fifth to one eighth pound per square inch per foot of column height. A minimum of three to four cubic feet of fluidizing gas (at conditions prevailing in the column) per 100 pounds of catalyst are required."

Supported Iron Catalysts

The ready availability of iron and its consequent low cost have long stimulated research on its use as a Synthine catalyst. However, the effect of catalyst costs on the ultimate cost of Synthine products is said to be relatively slight in the case of modern commercial cobalt complex catalysts,[234] and perhaps the greatest present interest in iron catalysts centers around their ability to produce more unsaturated (hence higher-octane) gasoline fractions and the possibilities they afford of utilizing water gas directly rather than after adjustment of the hydrogen-carbon monoxide ratio. An equimolar mixture of hydrogen and carbon monoxide is suitable for the synthesis.[26]

Alkali-promoted iron-copper catalysts were used by Fischer and Tropsch in many of their early experiments.[204] However, they found these catalysts to be markedly inferior to cobalt or nickel catalysts, both in product yield and length of life.[195] The use of alkali, besides increasing the yield of primary product, is said to aid in the production of solid paraffin wax. Copper may be added to precipitated or impregnated iron catalysts to facilitate the reduction step in their preparation and to favor adsorption of the alkali salt.[132, 195] By using less copper in the catalyst, one pair of investigators[189] claimed to have obtained improved results, although others[215] found little difference in the activities of catalysts with iron-copper ratios of 1:2, 1:1, 3:1, and 5:1. The latter observed that catalysts containing 0.5 percent alkali possessed longer lives than those containing greater proportions.

The promoting effect of alkali addition to iron catalysts is believed to be the result of the formation (in its presence) of cubic ferric oxide (Fe_2O_3) and the prevention of its transition to the less active magnetic iron oxide (Fe_3O_4).[218] A number of X-ray diffraction and magnetic susceptibility studies have been made of such catalysts,[26, 182, 219] more recent investigations[237] having indicated that the inactive catalyst contains β-FeOOH and the active catalyst contains either α-FeOOH or α-Fe_2O_3 or both, but no β-FeOOH. It was found[237] that the highly active catalysts had higher magnetic susceptibilities and

were ferromagnetic, a suggested explanation being the possible presence of ferromagnetic alkali ferrites in the active catalyst.

Catalysts prepared by impregnating asbestos with the nitrates of the catalytic metals and small amounts of rare earth oxides have been found to give the following yields per 1000 cubic feet of synthesis gas: Fe-Cu-Ni (4 to 1 to 0.01), 1.8 pounds; Fe-Cu-Ni (4 to 1 to 0.02), 2.8 pounds; and Fe-Cu-Ni-CeO_2-ThO_2 (4 to 1 to 0.05 to 0.001 to 0.006), 5.0 pounds.[206] Yields of 5.2 and 5.5 pounds per 1000 cubic feet of "water gas" ($1H_2$:1CO), respectively, were obtained with precipitated catalysts containing (in the first case) Fe, Cu, kieselguhr, and K_2CO_3 in the ratio 4 to 1 to 5 to 0.08 and (in the second case) the same ingredients plus 0.08 part of manganese.[222, 248] The addition of 5 percent of nickel or cobalt to these catalysts increased the yields when synthesis gas ($2H_2$:1CO) was used, but proved undesirable for the gas containing equal amounts of hydrogen and carbon monoxide.

Sintered Iron Catalysts

Partial sintering of precipitated iron-alumina catalysts by reduction at about 1550° F. has been patented by I. G. Farbenindustrie A. G.[262, 263, 266] This company also has patented catalysts of the iron group prepared by heating the corresponding metal carbonyl at a temperature above 930° F., but below the melting point of the metal, for such a period of time that partial sintering occurs.[264, 271, 272, 284] Earlier I. G. patents[257, 258, 259, 278] had specified the use of iron-group carbonyls in the synthesis under conditions which might possibly have caused some sintering.

In addition to use in the normal vapor-phase type of operation, such sintered-iron catalysts may be employed in liquid-medium operations.[267, 282, 283] They also may be made in pellet form by mixing iron powder with an alkaline metal salt solution (such as aqueous KCl), compressing, and reducing with hydrogen at about 1550° F.[268] Although single-pass yields are low, a satisfactory ultimate yield may be obtained by recycling the partially-spent synthesis gas.[286]

An I. G. Farbenindustrie report[26, 181], written in 1939, concerns a rapid, hot-gas-recycle process over pelleted, sintered-iron catalyst. The gas was passed through the reactor at hourly space velocities of 5,000-35,000, and the heat evolved in the synthesis was absorbed in an external heat exchanger. The reaction was conducted at 605-660° F. and 295 psi. The conversion was about 1 percent per pass, and about 1 percent of fresh synthesis gas ($0.9\ H_2$:1 CO) was added per cycle, an equivalent amount of recycle gas being removed, freed of carbon dioxide, and sent to a second, smaller (¼-size) recycle system. Conversion in the first stage was about 78 percent and in the second stage about 13.5 percent. The product was highly unsaturated, consisting mainly of gasoline of 85 Research octane number. A polymer gasoline of 97 O.N. was made from the C_2-C_4 unsaturates. The yield of primary product was about 10 pounds per 1000 cubic feet of synthesis gas, of which approximately 0.87 pound contained oxygenated compounds.

The hydrocarbons synthesized over sintered-iron catalysts consist largely of branched-chain paraffins, while those produced on non-sintered-iron catalysts contain very little iso-compounds. It is considered likely that sintering produces a critical spacing of the metal atoms in the iron carbide lattice which is conducive to the production of isoparaffins.[133]

Ruthenium Catalysts

Ruthenium or ruthenium-containing contact catalysts have been patented for the synthesis of solid hydrocarbons at temperatures of 300-540° F. and high pressures (over 450 pounds per square inch).[275] Studies of other Group VIII metals (rhodium, palladium, osmium, iridium and platinum) have shown that ruthenium is most satisfactory for this purpose.[227] The ruthenium catalyst is long-lived (no appreciable change in activity occurred during one six-months test) and gives yield of approximately 6.2 pounds of paraffin wax and 3.1 pounds of liquid per 1000 cubic feet of synthesis gas at 380° F. and 1500 psi.[197]

The effect of pressure upon the yield and type of products is very pronounced. At atmospheric pressure, the yield is very low and the product consists of liquid hydrocarbons.[196] As the pressure is raised, the conversion at 355° F. increases rapidly until about 4400 psi then more slowly above this pressure, about 4.7 pounds of solid paraffin per 1000 cubic feet of synthesis gas being produced at 1300 psi, 6.2 pounds at 4000 psi, and 7.2 pounds at 4400 psi.[277, 228] At all pressures, solid paraffins amount to about 60 percent of the total yield of liquid and solid hydrocarbons, and gaseous hydrocarbons constitute 25 percent of the over-all conversion. Fischer and Pichler[203, 227, 228] also studied the effect of pressure upon the synthesis over ruthenium catalysts, but they investigated only the range up to 1500 psi.

The use of ruthenium catalysts for the commercial production of high-melting wax is not considered feasible by Dr. Martin, director of Ruhrchemie A.-G., because of the relative scarcity of ruthenium. He has stated that hard wax can be produced more economically by using the conventional cobalt catalyst at low space velocities and low temperature.[26]

Effect of Reaction Temperature

The Synthine reaction takes place in a fairly narrow temperature range. Even with the most active nickel or cobalt catalysts, the reaction is very slow below 350° F. Above 440° F., with the same catalysts, the yield of liquid hydrocarbons diminishes rapidly, and methane yields increase correspondingly.[241] At higher temperatures (575-660° F.), methane production predominates, and the oxygen in the synthesis gas appears in the product as carbon dioxide rather than water. As mentioned, the optimum operating temperature with iron catalysts is higher than with nickel or iron, being about 465° F. However, the range in which satisfactory results are obtained is also quite small. The temperature coefficient of the synthesis on cobalt and iron catalysts is about 1.6 per 18° F. in the range of 370° to 445° F.[237] While the possibility of obtaining a given type of product depends to a large extent upon the catalysts and pressure employed, it is known that, in general, the hydrogenation of carbon monoxide produces straight-chain hydrocarbons at relatively low temperatures, alcohols in the range 575-750° F., isoparaffins in the range 750-885° F., and aromatics in the range 885-930° F.[26, 208]

Effect of Pressure

Generally speaking, the use of higher pressures in the Synthine process favors the formation of oxygenated compounds and hydrocarbons of high molecular weight, thus reducing the yield of liquid hydrocarbons.[195, 198] However, the use of medium pressures 75-220 psi) has definite advantages, both in its effect on the total yield and upon the composition of the

TABLE 1
Effect of Pressure on Yield and Type of Synthine Product[201]

PRESSURE, psig.	YIELDS, POUNDS PER 1000 CUBIC FEET OF SYNTHESIS GAS				
	Total, Solid and Liquid	Paraffin Wax	Liquid, Above 390 °F.	Gasoline, Below 390 °F.	Hydrocarbon Gases, C_1-C_4 Inclusive
0	7.28	0.62	2.36	4.30	2.37
22	8.16	0.93	2.68	4.55	3.12
73.5	9.35	3.74	3.18	2.43	2.06
220	9.03	4.36	2.24	2.43	2.06
735	8.59	3.36	2.30	2.93	1.31
2200	6.48	1.68	2.12	2.68	1.93

product. Using a cobalt-thoria-kieselguhr catalyst without revivification over a period of one month, Fischer and Pichler[201] found that (1) the total yield increased with increasing pressures up to about 75 pounds per square inch; (2) the yield of paraffin wax increased with pressure up to about 220 pounds per square inch; and (3) the active life of the catalyst was greatest when medium pressures were used. Table 1 shows the results of their experiments over a pressure range of 0-2200 pounds per square inch. Martin[220] has reported similar results in two-stage operations.

At pressures of 75-220 pounds per square inch, the total yield of liquid and solid hydrocarbons obtained by Fischer and Pichler in the experiments just mentioned amounted to 9.35 pounds per 1000 cubic feet of synthesis gas when fresh catalyst was used and still exceeded 6.24 pounds after 26 weeks of operation without catalyst revivification. On the other hand, at atmospheric pressure, the initial yield was 8.11 pounds and declined to 6.24 pounds after only one month of operation.[201] The effect of pressure on the synthesis over ruthenium catalysts has already been discussed.

The use of moderate pressures also offers the practical advantage of reduction in plant size for a given output.[129]

Effect of Throughput Rate

Using a cobalt-thoria-kieselguhr (100:18:100) catalyst in two-stage operation at a pressure of 150 pounds per square inch, Fischer and Pichler[202] found that both the yield of solid paraffin and the total yield of higher hydrocarbons decreased with increasing rate of throughput. At a throughput of 3.2 cubic feet per hour per pound of cobalt in the catalyst, they obtained yields of about 11.8 pounds of primary product per 1000 cubic feet of synthesis gas, of which 48 percent consisted of solid paraffin; 44 percent, liquid hydrocarbons; and 8 percent, C_3-C_4 hydrocarbons. When the throughput was increased to 32 cubic feet per hour, 9.0 pounds of primary product were obtained, of which 14 percent was solid paraffin, 73 percent was liquid hydrocarbons, and 13 percent was C_3-C_4 hydrocarbons. The necessity for utilizing higher reaction temperatures at increased throughput rates in order to maintain good yields leads to the formation of more methane. If the temperature is held constant, the yield drops rapidly with increased throughput rate, as may be seen from Table 2, which shows the results obtained by using a cobalt catalyst at 390° F. and 220 psi.

Similar data have been reported by Japanese[239] and British[179] investigators. Working at lower throughputs than those of Table 2, Tsuneoka and Nishio[249] have observed that the yield rises to a maximum with increasing throughput before falling off. High space velocities favor the production of olefins.[132]

Generally speaking, "reducing the space velocity has an effect on the products similar to increasing the pressure, but with the important difference that a low space velocity reduces the output of the plant and a high pressure increases it."[129]

Effect of Hydrogen-Carbon Monoxide Ratio

As might be expected, increasing the relative amount of carbon monoxide to hydrogen in the synthesis gas results in the formation of a more olefinic product and the production of more carbon dioxide.[255] A high hydrogen-to-carbon monoxide ratio produces a saturated product and favors high methane formation. The amount of olefins in the reaction product has been found to be inversely proportional to the hydrogen content of the synthesis gas.[228] With conventional catalysts, a ratio of $2H_2$ to 1CO is considered optimum for obtaining the maximum total yields of hydrocarbons.[239]

German Commercial-Scale Process

The reports of the Combined Intelligence Objectives Subcommittee (CIOS) and the United States Technical Oil Mission (TOM), both of which investigated German technology in the closing days of the war and for a time thereafter, offer a fairly complete picture of the German process for the commercial-scale hydrogenation of carbon monoxide. The German synthesis plants operated at temperatures of 355-390° F., either at atmospheric pressure or at medium pressures of about 150 psi. The choice of atmospheric or medium-pressure operation, in any given plant, was determined largely by the products desired. The medium-pressure synthesis is said to have given three times as much solid paraffin (from which higher alcohols can be made) and about two-months-longer catalyst life periods than were obtained in normal-pressure operations. However, the synthesis at atmospheric pressure gave a higher gasoline yield. Martin (Ruhrchemie) believes that any postwar plants built in the predictable future should be of the medium-pressure type (because of the synthetic chemical possibilities), since the (German) synthetic gasoline (from coal) will not be able to compete with that made from petroleum, assuming normal peacetime conditions and a free world economy.[26]

The catalyst ovens used in the middle-pressure synthesis are interesting pieces of equipment, since they appear adaptable to almost any exothermic catalytic process carried out at constant temperature: e.g., the methanization of coke-oven gas.[17,115] These ovens consist of groups of vertical tubes of two sizes, the smaller tubes being placed inside the larger ones, concentrically. The catalyst is placed in the annular space between the tubes. Temperature control during the exothermic reaction is maintained by passing steam through the inner tubes and the whole of the space between the outer tubes. Another type of medium-pressure reactor, the "Taschen-Rohr Ofen" (Chamber-Tube Oven), consists of single tubes containing a complex web of plates to facilitate the dissipation of heat.[17,115]

The synthesis was carried out in two or three stages, with product recovery after each stage.[26,413] On the average, about 9.4 pounds of primary product

TABLE 2
Effect of Increased Throughput on Yields at Constant Temperature[201]

Throughput (cu. ft./hr./lb. cobalt	18.4	37.0	57.6	160
Total Yield (lbs./1000 cu. ft. syn. gas)	6.30	5.30	3.74	1.03

were obtained per 1000 cubic feet of synthesis gas. The constituents of the primary product ranged from propane-propylene to waxes of molecular weights as high as 2000.[237] Generally speaking, the yield and quality of the products from the wartime synthesis did not differ materially from prewar published information. Olefins of the C_3-C_4 fraction were converted to alcohols, etc., and residual paraffins were used as motor fuel. Low-boiling-range gasoline of low octane number was used for blending in motor fuels. The middle fractions of high cetane number were used as diesel fuel. The paraffin wax was largely converted into fatty acids for soap manufacture. Lubricating oils were synthesized from the heavy fractions.[26]

Considerable experimental work was done during the war on the development of recycle processes for the synthesis over commercial cobalt catalyst.[26] In contrast to the previously-mentioned rapid recycle process studied by I. G. Farbenindustrie for use with its sintered-iron catalyst, these processes utilized a relatively low recycle ratio and the familiar, externally-cooled, middle-pressure reactor. On the pilot scale, Lurgi-Metalgesellschaft found it possible, at a recycle ratio of 3:1, to increase the throughput by 30 percent without a sacrifice in yield or to realize a yield of about 10.6 pounds per 1000 cubic feet of synthesis gas at normal throughput. On the basis of pilot-plant performance, conversion of two commercial-scale plants to such operation was reportedly planned. Ruhrchemie A.-G., after similar experiments on the laboratory scale, installed recycle equipment in its Sterkrade plant, but this was destroyed by bombing before it could be put into operation.[26]

Iso-Synthesis Reaction

The conventional Synthine process, whether conducted at atmospheric or medium pressures, produces only small quantities of branched-chain hydrocarbons. The isobutane-isobutylene content amounts only to about 10 percent of the C_4 fraction with a cobalt catalyst and to about 15 percent with an iron catalyst. The concentration of branched-chain compounds rises to approximately 20 percent in the C_8 fraction with both catalysts, and the C_{16} fraction consists exclusively of isocompounds. However, the extent of branching seldom exceeds one methyl group on a long chain.[207, 208]

Although it has, as yet, been operated only on an experimental scale, the "Iso-Synthesis" process is of great interest since it offers means for producing high-octane gasoline (via alkylation) by a modified form of the Synthine process. It might be nearer the truth to say that the Iso-Synthesis is a modification of the Synthol process, rather than of the Synthine process, since it actually consists of first forming "higher" alcohols (mainly isobutanol), then dehydrating the alcohols to olefins, and finally hydrogenating the olefins to the corresponding isoparaffins. The process combines these three reactions in a single stage. By reacting synthesis gas containing about 1 part hydrogen to 1.2 parts carbon monoxide over catalysts consisting of ZnO, Al_2O_3, ThO_2, or mixtures of one with another at temperatures of about 840° F. and pressures of about 4400 pounds per square inch, over 90 percent concentrations of isoparaffins have been obtained in the C_4 and C_5 fractions of the reaction product.[190, 207, 208]

Research on the Iso-Synthesis reportedly was begun at the Kaiser Wilhelm Institut für Kohlenforschung in the fall of 1941[207], and mention of it was made by Fischer[194] in 1943. However, no details were made public until after the investigation of the institute by the U. S. Technical Oil Mission.[208] According to Dr. Helmut Pichler, the acting director of the institute, the catalysts suitable for the reaction include: (1) zinc oxide with alumina, (2) thoria alone, (3) thoria with alumina, (4) alumina alone, and (5) zinc oxide with thoria, cerium oxide, or zirconium oxide. The rate of throughput is five to ten times greater than that used in the conventional Synthine process. The yield of C_4 and higher hydrocarbons ranges from 3.74 to 6.85 pounds per 1000 cubic feet of the synthesis gas, depending upon the catalyst used. Typical product distribution in the C_4 and higher fraction is as follows:

C_4H_{10} (90 percent isobutane)	60-70 percent by weight
C_5H_{12} (96-98 percent isopentane)	20-30 percent by weight
C_6-C_8 compounds	Small amounts

The C_6 fraction contains mainly 2-methylpentane and 3-methylpentane, but no neohexane or normal hexane. Some naphthenes have been found in the C_6-C_8 fraction. Larger quantities of hydrocarbons boiling above C_5 are obtained at lower reaction tempertures. About 0.62 pound of propane per 1000 cubic feet of synthesis gas is formed under the usual reaction conditions. In addition to the hydrocarbons produced, some alcohols, amounting to less than 10 percent of the total hydrocarbon yield, appear in the reaction product. These alcohols consist primarily of isobutanol and a little methanol. The tail gas contains about 10 percent methane (maximum), 30 percent carbon dioxide, 20 percent nitrogen, and 40 percent carbon monoxide-hydrogen.

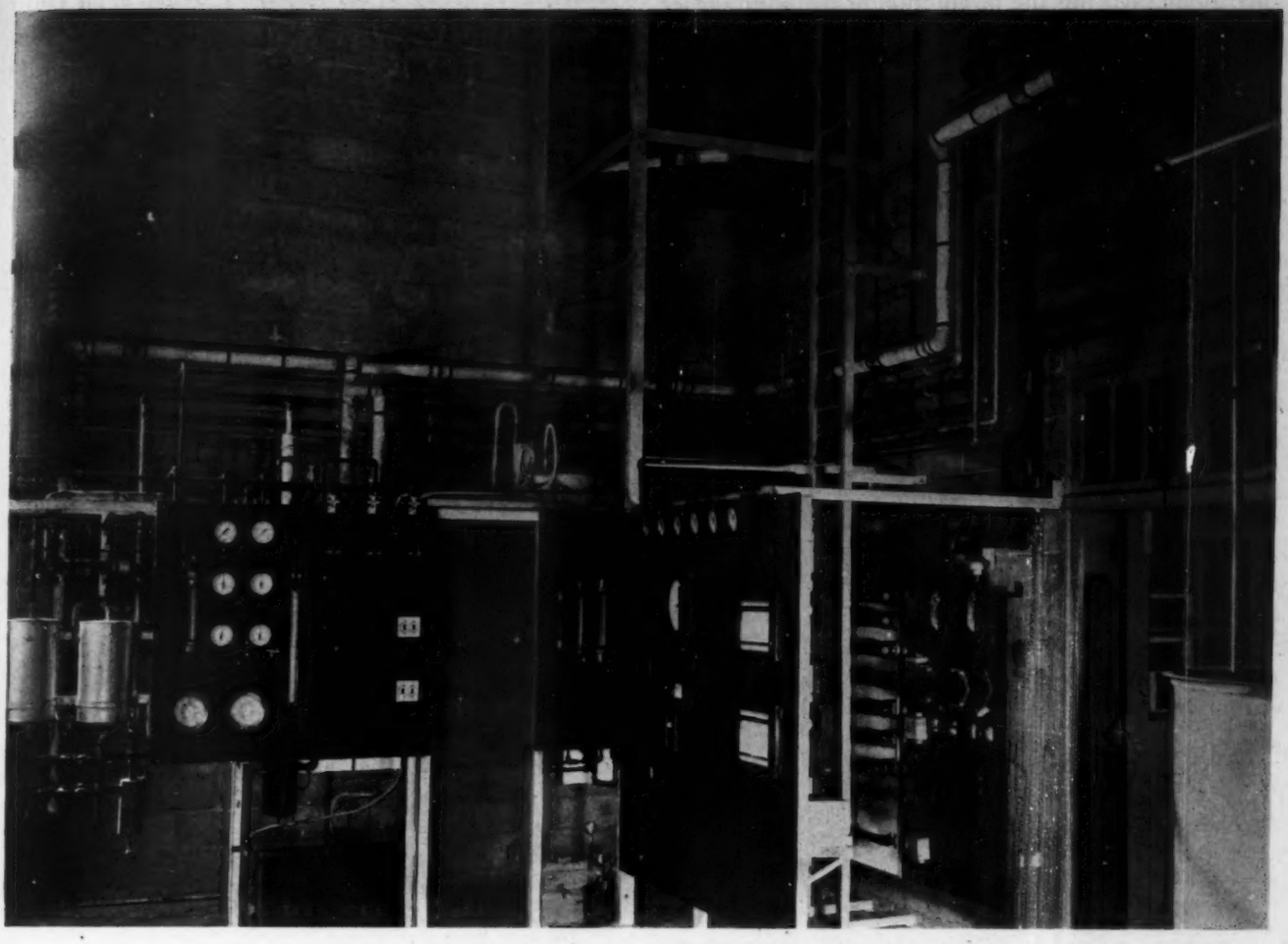
The temperature of operation is said to have a marked effect upon the reaction. At 750° F., alcohol production is high; at somewhat higher temperatures, dimethyl ether is formed; and around 840° F., isobutane is the major product. Raising the temperature still higher results in excessive carbon formation, and the catalyst requires frequent regeneration.

Carbon deposition puts a limitation on the size of the reaction tubes which may be used. Tubes of 12-15 millimeters (roughly half-inch) in diameter have proved satisfactory, while larger tubes have been found to encourage excessive formation of carbon.

Operation at pressures above 4400 pounds per square inch results in increasingly greater yields of oxygenated compounds. At 14,700 psi, the principal product of the reaction is dimethyl ether. On the other hand, pressures below 4400 psi result in smaller total yields.[190]

Aromatization Reaction

The Kaiser Wilhelm Institut was also engaged in the investigation of a process for producing aromatics by the hydrogenation of carbon monoxide.[26, 190, 194, 208, 233] Fischer[194] hinted at this work in 1943, and on-the-spot interrogation of Pichler[208] supplied what details are known. The reaction is reportedly conducted over a catalyst consisting of an oxide of chromium, molybenum, or thorium at a pressure of about 440 psi and temperatures ranging from 885-930° F. About 5-10 percent of potassium carbonate is included in the catalyst to reduce carbon formation. A 1:1 ratio of hydrogen to carbon monoxide is used.[26] The yield of liquid hydrocarbons is very small, usually below 0.62 pound per 1000 cubic feet of synthesis gas, but the product contains about 50 percent aromatics (toluene and xylenes), the remainder consisting largely of naphthenes.[208, 233] The major portion of the synthesis gas is converted to methane and car-



Courtesy H. H. Storch, U. S. Bureau of Mines

Internally-cooled experimental Synthine plant of Bureau of Mines.

bon. This synthesis was not considered very promising by the Germans.[190, 233]

Synthol Process

As mentioned in Article I, one domestic engineering concern[121] has recently announced that it is prepared to erect hydrocarbon synthesis plants which would use the "Synthol" process, developed as the outgrowth of original research and German data. No technical details were given, but it was mentioned that "this Synthol process * * * under controlled conditions produces various chemical products in the form of oxygenated compounds." It was also stated, as is common knowledge, that "changing the operating conditions will control the quantity and quality of the production of the hydrocarbons and of the chemical compounds."

It has been noted that the German "Synthol" is a mixture of hydrocarbons and (chiefly) oxygenated compounds, produced by high-pressure synthesis, and that this use of high pressure materially reduces the liquid hydrocarbon yields.[198] Whether or not this new "Synthol" process is similar in nature to its German namesake has not been revealed, but it would be no very difficult feat to produce hydrocarbon products from such a mixture as the German "Synthol," perhaps by hydrogenation or even by a reaction related to hydroforming.

As is evidenced by the commercial process for the production of synthetic methanol, oxygenated compounds can be produced in good yields by the hydrogenation of carbon monoxide over appropriate catalysts and under proper conditions (high pressures). Space does not permit a detailed analysis of these reactions here, although they most certainly constitute part of the prior art of the Synthine process.

Methane Synthesis

Considerable interest has been evidenced abroad in the production of methane from carbon monoxide and hydrogen, although this is a reaction usually minimized in every possible way during the normal Synthine process. "Synthetic natural gas" has a much more desirable calorific value than the water gas from which it is produced, so that this phase of the Synthine process constitutes a "gas-upgrading" method. Similarly, the other hydrocarbon gases produced to a greater or lesser extent—ethane through the butanes—are desirable fuel gases themselves or may be used for gas enrichment. Attention to these matters has also been given in this country.[90]

Gerdes[205a] has given this matter some attention in an article published abroad in 1941, and a recent British article[186a] has dealt with the subject in some detail. Reference should also be made to the section of this article on the reaction mechanism; where methane is desired as the primary product, every effort is made to avoid polymerization of the (CH_2) groups which form on the hydrogenating catalyst. Logically, nickel is the basic material of most of the catalysts investigated.

Komarewsky and Riesz[90] have reported that "the

conditions favoring the production of methane rather than Fischer-Tropsch (Synthine) synthesis of higher-molecular-weight hydrocarbons are: (1) catalyst, (2) relatively high temperature, and (3) high proportion of hydrogen in the reacting gas. Nickel and iron are active at atmospheric pressure. The temperature required is about 570° F. for nickel catalysts. The ratio of hydrogen to carbon monoxide theoretically should be three ($CO + 3H_2 \rightarrow CH_4 + H_2O$)." Even CO_2 can be hydrogenated to methane under certain conditions.

* * *

The remaining articles in this series will deal with the products and by-products of the Synthine process and with its economics and current status. Intense interest continues to be evidenced in the commercial application of the process in this country, and it seems likely that the date of initial plant operations is not many months removed, since at least one plant has already been announced.

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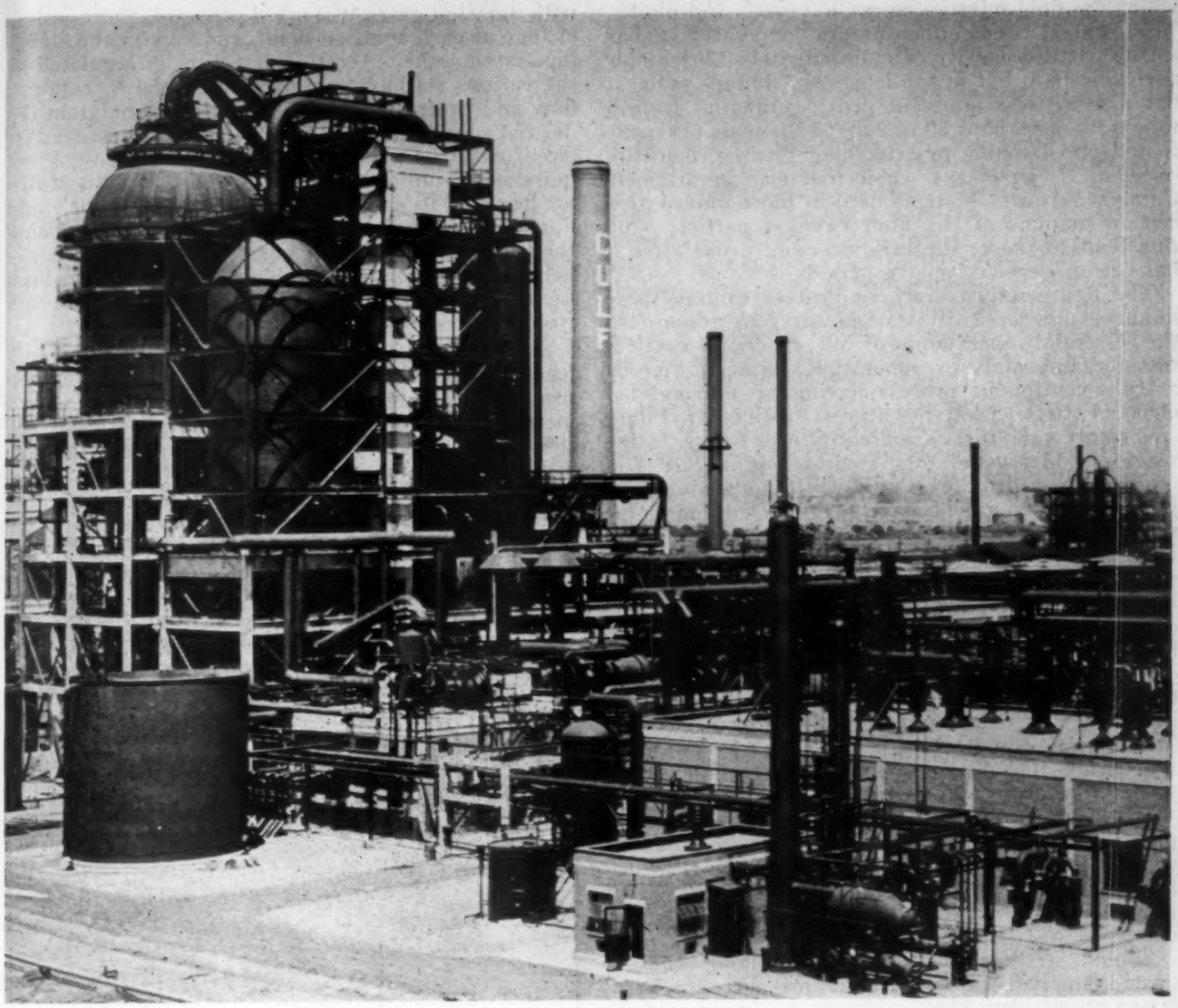

Fluid-Catalyst Technique

L. S. DANIELS, Associate Editor

THE "fluid" catalyst cracking units built in many refineries during recent years have introduced a new catalysis technique in refinery process operations. The catalyst utilized in these plants is in powder form, and may be either "natural" or "synthetic." Natural catalyst is excavated from naturally occurring clay deposits and ground to the desired particle size range, while synthetic catalyst is a manufactured material of similar chemical composition which possesses some advantages from the process standpoint. Particle size of the catalysts range between zero and 100 microns, the bulk being between 20 and 40 microns in most cases. (A micron is 1/1000 of a millimeter.)

Mechanics of Flow

If suitable amounts of gas (either steam, air, or oil vapor) are mixed with this powdered catalyst, the mixture becomes "fluidized" and can be handled much like other fluids. The fluid density can be controlled as desired, within limits, by varying the amount of gas added and by changing the rate at

which the fluid mixture moves in vessels and piping. The catalyst used, either natural or synthetic, has a true particle density of approximately 130 pounds per cubic foot, but in the loose, or "fluffed" state, it has a much less apparent density, ranging from a minimum of perhaps 30 to about 55 pounds per cubic foot. Upon tamping or after long settling, densities of 40 to 75 pounds per cubic foot may be attained. Catalyst recovery systems used in these units lose a certain amount of the finer catalyst particles and this tends to make the catalyst more dense after it has been in service for long periods.

The fluid catalyst cracking process utilizes these fluidized powdered catalyst mixtures to accomplish the essential operations of oil reaction, catalyst transfer, and catalyst regeneration, achieving thereby a truly continuous process having no moving mechanical parts within the process equipment. Fluidized catalyst mixtures are used in three forms. In the reactors and regenerators, indicated in Figure 1, a more or less definite "liquid level" is maintained, the catalyst being held up in a "fluid" bed. Standpipe aeration gas, plus oil vapor and steam for the reactor and combustion air for the regenerator, plus catalyst, enter these vessels at the bottom and mix into the turbulent fluid bed. The major part of the catalyst settles out here and the gases flow up through the catalyst and leave at low velocities, the order of magnitude being 1 to 2 feet per second. At these velocities the amount of catalyst entrainment is not serious. The catalyst bed is turbulent, with little variation in temperature or channeling of aeration gases. Bed densities may range from 15 to 35 pounds per cubic foot, normally are between 25 and 30 pounds per cubic foot, and are termed "dense phase." Catalyst leaves the vessel at the bottom, by gravity flow, and is permeated with vapors which fill the voids between catalyst particles to the extent required by the apparent or "flowing" density of the catalyst.

"Standpipes" are used to build up fluid heads to provide the necessary differential pressures for circulation of catalyst. Flow of catalyst is downward by gravity, as indicated in Figure 2. Aeration gas must be provided to set up a fluid condition in the standpipe. With a static bed, or with low-velocity standpipe flow, the aeration medium rises upward through the catalyst, just as in the case of the reactor and regenerator catalyst beds. When standpipe velocities exceed approximately 2 feet per second, the aeration gases will be carried downward along with the catalyst particles. This type of flow is also termed "dense phase." The pressure at any given elevation in a standpipe, reactor, or regenerator "dense-phase" bed is equal to the total catalyst head above that point plus the pressure existing in the equipment above the catalyst bed. Standpipe densities range from 15 to 30 pounds per cubic foot, and are normally between 25 and 30 pounds per cubic foot. At the bottom of the standpipe pressure is higher than at the inlet end, so that the aeration gas is compressed and a greater quantity is required to maintain a given flowing catalyst density than is necessary higher up the standpipe. To supply this gas, standpipes are provided with aeration-injection points at about 15-foot vertical spacing. At low velocities of standpipe flow, with aeration gases rising upwards into the vessel above, there is a noticeable change in standpipe catalyst density at each aeration level, the amount of variation depending upon the quantity of gas injected at that point. At higher velocities, with aeration gases moving downward, there is less change at each aeration point, and flow in the standpipe is smoother. Aeration injection is regulated at the various stations to allow "dense-phase" catalyst, flow and control standpipe densities to maintain the desired standpipe fluid head for optimum catalyst circulation. In the calculation of aeration-gas requirements uniform standpipe densities usually are assumed to make certain that "dense-phase" fluid flow can be attained, but in actual plant operation the quantity of aeration gas to standpipes often is held to the minimum operable value since it has been found that less erosion of standpipe walls results from this procedure.

Standpipe design velocities run as high as 5 to 7 feet per second. The quantity of catalyst flowing from each standpipe is controlled by a "slide valve" located at the outlet end, the pressure drop across these valves serving to balance the other pressure differentials of the catalyst-circulation system.

Catalyst is elevated from low points in the catalyst system by means of "carrier" lines which utilize a flowing stream of gas as a carrying medium for the powdered catalyst. This type of flow, termed "dilute phase," is indicated in Figure 3. The energy to establish flow in this case comes from the pressure level of the carrier medium and the fluidized catalyst, rather than from the attraction of gravity, so that flow in an upward direction is possible, given suitable pressure differentials between inlet and outlet ends of the line. Minimum velocity at which this type of flow will occur is about 15 to 17 feet per second, and flow is much smoother between 20 and 30 feet per

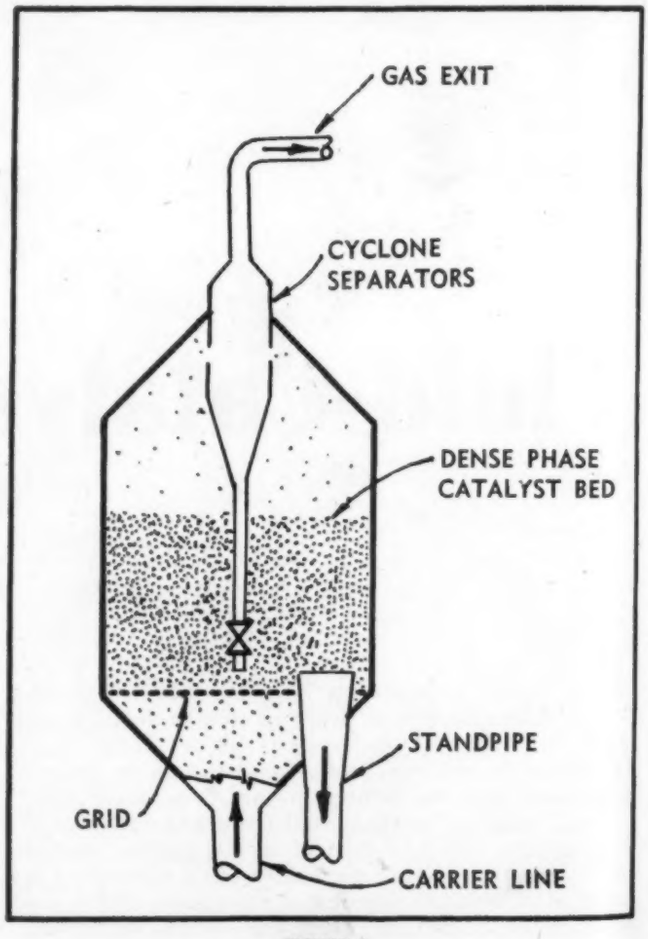

FIGURE 1

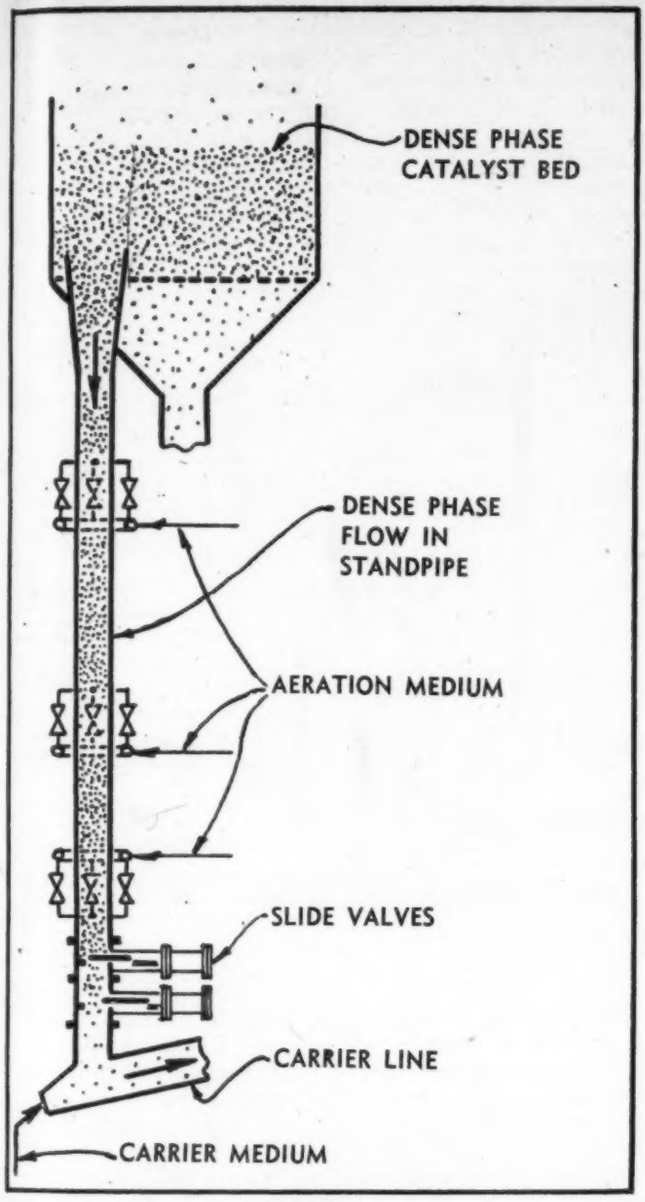

FIGURE 2

second. As velocity is increased, erosion of piping becomes serious, with 40 feet per second possibly an excessive rate. The carrier gas moves at a higher velocity than the catalyst particles in this type of flow. The actual amount of "slip" is difficult to evaluate, being dependent on particle size, amount of carbon on catalyst, velocity, and other variables, but because it has marked influence on flowing density in carrier lines, some allowance must be made. Usual design practice has been arbitrarily to set slip at 2:1, that is, to multiply by 2 the density calculated from gas and catalyst flow quantities when estimating the "dilute phase" head differential of the carrier line. Densities in carrier lines have been set as high as 5 pounds per cubic foot, slip neglected, for design purposes.

Typical Unit

In Figure 4, a simplified flow diagram of the catalytic section of a typical fluid cracking unit is shown, with flow quantities and process conditions used for a current motor-gasoline operation. Fresh feed, 28.5° API virgin gasoil from mixed Coastal crudes, with a boiling range from 300° F. initial to 740° F. 90 percent point, is pumped into the unit through heat exchange, where it reaches a preheat approximately 380° F., to the base of the regenerated catalyst standpipe where it is contacted with hot catalyst flowing down from the regenerator.

The feed, substantially liquid up to this point, is heated to about 850° F. by the catalyst, steam is injected lowering the partial pressure of the oil feed, and this, in combination with the elevated oil temperature, results in complete vaporization of the oil. The oil vapors, steam, and catalyst pass up the reactor feed riser in "dilute-phase" flow, the oil vapor and steam serving as carrier medium for the catalyst particles. The reactor inlet stream enters the reaction zone proper, the reactor "dense-phase" catalyst bed, through a "grid" distribution plate containing evenly-spaced orifice holes through which the entering material passes at increased velocity and with considerable pressure drop. This grid serves as a separating partition between the "dilute-phase"-inlet-line material and the "dense-phase" catalyst bed above, and prevents channeling and uneven flow. Reactor-product vapors and steam rise through the catalyst bed and leave at the top of the vessel through cyclone type separators which remove most of the entrained catalyst, returning it to the reactor catalyst bed through a "dip leg" pipe by gravity flow. Reactor products are fractionated in a primary distillation tower which makes a gasoline cut overhead, gasoil sidestreams, and a bottom slurry stream containing

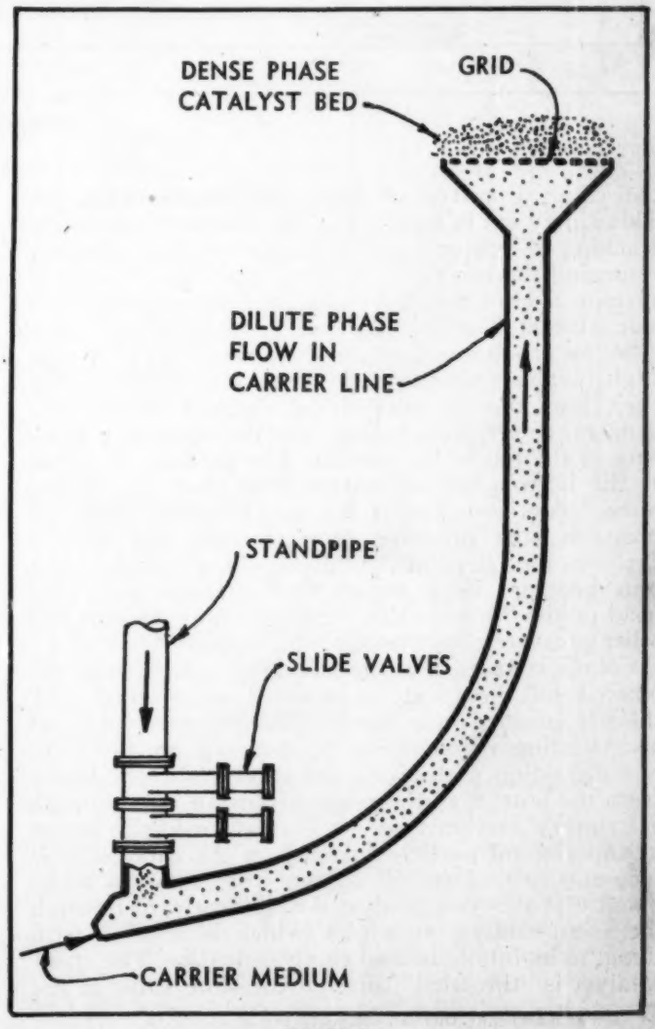

FIGURE 3

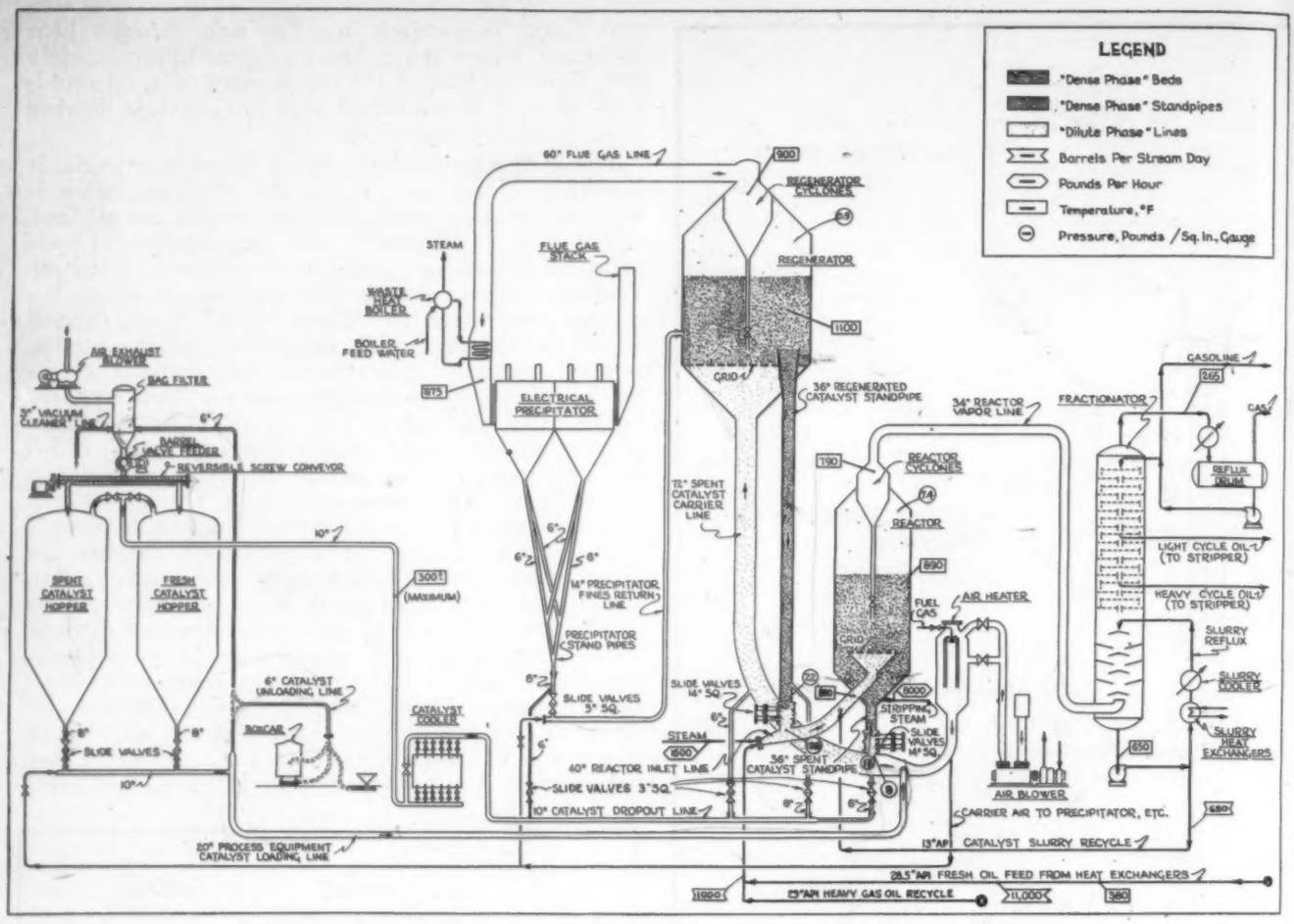

FIGURE 4

the catalyst carryover from the reactor cyclones. This slurry cut is recycled to the reactor for ultimate cracking to lighter liquid products and coke, thereby returning the carryover catalyst to the main catalyst system. Part of the heavy gasoil produced as a tower side stream also is recycled to the reactor. The gasoline product is condensed overhead, at pressures slightly above atmospheric. The reflux-drum pressure, plus the pressure drops through reflux condensers, tower, and piping sets the operating pressure at the top of the reactor. The pressure required at the bottom of the reactor feed riser for "dilute phase" flow to occur, is the sum of the reactor top pressure plus pressure drop through the reactor cyclones plus head of the "dense phase" catalyst bed plus pressure drop across the entrance grid plus head of the "dilute phase" catalyst plus friction and other pressure drop* in the inlet riser.

Coke produced by the cracking reaction is deposited on the catalyst particles, interfering with their continued use as a cracking catalyst, and necessitating regeneration by burning off the coke in combustion with air. Spent catalyst is withdrawn from the bottom of the reactor, flowing first through a stripping section where it is contacted with steam to vaporize oil particles carried on the catalyst surface and to displace oil vapors from catalyst voids. Spent and stripped catalyst flows downward through the spent-catalyst standpipe which is aerated with steam to maintain desired catalyst density. The spent catalyst is "throttled" through the slide valve at the bottom of the standpipe and meets a carrier stream of air which transfers it upward in "dilute-phase" flow to the regenerator which it enters through a distribution grid, this carrier stream also serving as combustion air for burning coke from the catalyst. Some combustion starts on contact between spent catalyst and air, since the catalyst is well above the ignition temperature of the coke, but the major part occurs in the "dense-phase" catalyst bed in the regenerator where the catalyst is held at combustion conditions long enough to insure adequate regeneration. In this particular unit, combustion flue gases are exhausted to atmosphere through cyclone separators, a waste-heat boiler, and a Cottrell type electrical precipitator, with no back-pressure valves in the vent system, and the regenerator pressure is very slightly above atmospheric. The electrical precipitator recovers a large percentage of the catalyst carryover from the cyclones which is returned to the regenerator by means of standpipes, a slide valve, and carrier air system. For the spent-catalyst carrier line, as in the similar case of the reactor feed riser, the pressure required at the inlet for "dilute-phase" flow to occur is the sum of the regenerator gas outlet pressure plus pressure drop through the regenerator cyclones plus head of the "dense-phase" catalyst bed plus pressure drop across the entrance grid plus head of the "dilute-phase" catalyst plus friction and other drop in the carrier line.

The pressures required to allow catalyst to flow into the inlet of the reactor feed riser and the inlet of the spent-catalyst carrier line are "built up" by

* Flow around bends, through enlargements, etc.

catalyst standpipes from the regenerator and reactor, respectively. Since the reactor top pressure in this particular case is about 7 psi higher than the regenerator top pressure, and the other pressure heads are comparatively less or tend to balance each other, a greater height of standpipe is required above the inlet to the reactor feed riser than above the inlet of the spent-catalyst carrier line, and the regenerator is elevated a considerable distance above the reactor as a result. In this unit a maximum reactor pressure considerably above the 7 psig shown in Figure 4 can be attained by the catalyst-circulation system since the reactor is located high enough to provide necessary standpipe head at the bottom of the reactor-feed riser. Unbalanced pressure differentials in the catalyst-circulation system are absorbed in pressure drops across the slide valves at the base of each catalyst standpipe. To maintain uniform reaction conditions it is necessary to hold a steady volume of catalyst in the reactor, and the slide valve at the bottom of the spent-catalyst standpipe is controlled, indirectly, to maintain the desired catalyst inventory in this vessel. Variations in total inventory of catalyst resulting from catalyst losses and fresh catalyst additions are reflected in the amount of catalyst present in the regenerator. Within the fairly wide requirements of satisfactory coke combustion, the regenerator catalyst quantity can be varied as may be convenient. As protection against shutdown from erosion, two slide valves in series are provided at the bottom of each standpipe. One valve is kept open, normally, with one valve in service holding the necessary pressure drop. When a slide valve becomes too badly worn it is removed from service by being opened wide and its spare is controlled to maintain the pressure drop. In plant operation standpipe densities and catalyst-system pressures usually are adjusted so that minimum pressure drop occurs across the slide valves, as this greatly lessens erosion.

Heat Balances

The powdered catalyst used in fluid catalytic cracking units, in addition to serving as catalyst for the cracking reaction, and as vehicle for transferring coke production from the reactor to the regenerator, also serves as a direct-contact heat medium for vaporizing the oil charge, heating it to cracking temperature, and supplying the heat of reaction. The unit shown in Figure 4 uses hot catalyst circulation plus heat exchanger to supply all process heat. In such a unit it is necessary to balance heat requirements of the reactor system against the heat supplied by the regenerated catalyst circulation. Several operating variables are interrelated, and one can not be changed without having some effect on the others. The reactor temperature and reactor catalyst inventory are dependent on the nature of the oil charge, the desired products, and the catalyst activity. The regenerator temperature must be sufficiently high to promote coke combustion, but not high enough to cause catalyst deactivation. The catalyst circulation rate, the differential between reactor and regenerator temperatures, and the feed preheat must all be adjusted so that the desired heat balance is attained. The weight ratio of catalyst entering the reactor to the reactor oil charge including any recycle oil is termed the catalyst-oil ratio. This ratio is about 12:1 for the conditions shown in Figure 4 which involve a fairly low reactor temperature and moderate conversion. More severe cracking conditions with higher reactor temperature and higher conversion will require a much higher catalyst circulation rate, that is, a higher catalyst-oil ratio, to secure heat balance. Addition of a preheat furnace for the oil feed will provide operating flexibility and allow a lower catalyst-oil ratio by raising the total heat of the oil feed. At higher conversion levels with higher reactor temperatures, the lower differential temperature between regenerator and reactor and the greater heat required for the cracking reaction necessitates an increase in the catalyst-oil ratio. If no preheat furnace is used this ratio may be 25:1 or higher. At low conversion levels with low reactor temperature and with a preheat furnace, the catalyst-oil ratio may be as low as 5:1.

The coke-burning capacity of the regenerator is usually the basic limiting factor that sets maximum oil throughput for a unit, and this capacity is directly related to the rate at which heat can be removed from the regenerator "dense-phase" catalyst bed by circulation of catalyst to the reactor, by exit of flue gases from the regenerator, or by other means. The

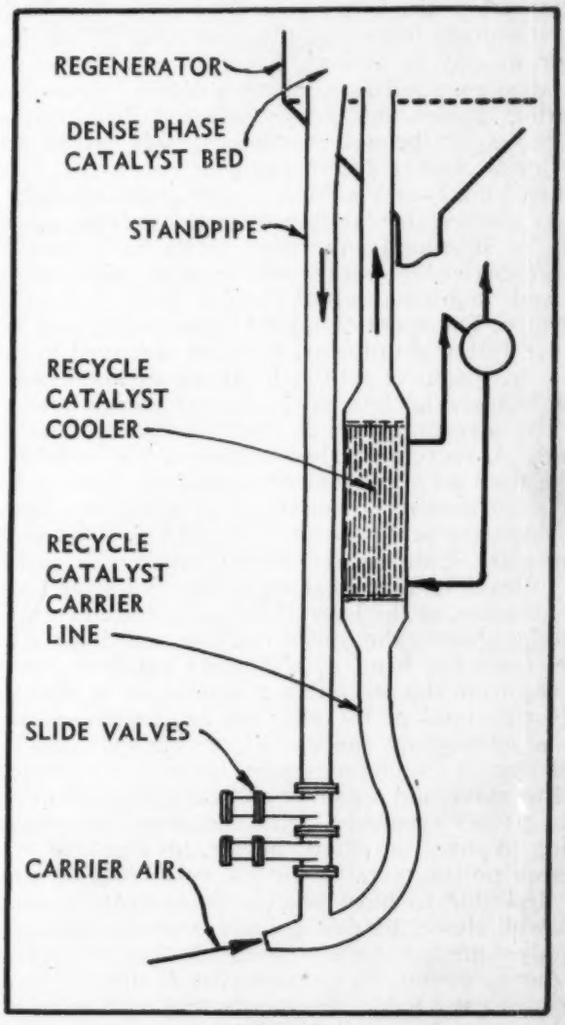

FIGURE 5

provision of a "recycle catalyst cooler" is one method for removing heat from the regenerator. As indicated in Figure 5, the cooler can be a tube bundle through which catalyst is circulated in "dilute-phase" flow by means of a special standpipe, slide valve, and carrier line. Air carrier medium is used in the regenerator for coke combustion. The heat removed from the recycled catalyst is possibly best utilized for

steam generation as there is a fire hazard from tube failures if oil is heated here.

Auxiliary Catalyst Equipment

The principal auxiliary equipment required for handling catalyst in a fluid cracking unit is shown in Figure 4. Fresh catalyst is brought to the refinery in box car quantities, either loose or packaged in sacks. An air conveyor system is used to move this catalyst into the storage hoppers. Air is drawn through a 6-inch pipe by means of a positive-displacement rotary compressor. The inlet to this line, a section of hose, is located at the railroad unloading site, and is either inserted into the loose catalyst, or if the catalyst is packaged, it is connected to an open hopper into which the sacks are emptied. The suction line passes through a separator with bag filters located above the catalyst storage hoppers which separate catalyst from airstream, collecting the catalyst in a chute. A rotating barrel-valve type feeder allows the catalyst to flow by gravity into a reversible horizontal screw convevor which moves the catalyst to the inlet opening of either the fresh-catalyst storage hopper or the spent-catalyst storage hopper, as may be desired.

Catalyst required for makeup purposes to replace operating losses can be transferred from either storage hopper through standpipes, slide valves, and a carrier air line to the regenerator.

When a fluid cracking unit is shut down it is necessary to remove the catalyst from the process equipment, as it would otherwise settle and pack in catalyst carrier lines and would interfere with inspection and maintenance, and with normal start-up procedure. The spent-catalyst hopper serves to store catalyst during shutdowns. Catalyst is moved to this hopper by means of a 10-inch carrier air line passing through a special box type catalyst cooler used to cool the stream to about 300° F. as it flows to storage. A recycle catalyst cooler, when available, can be used as a convenient means for cooling hot catalyst to storage temperatures at shutdown times, and makes the provision of a special box type cooler unnecessary. Catalyst removed to storage is picked up by means of the usual standpipe and slide valve arrangements, at the base of the regenerated catalyst standpipe, below the spent catalyst standpipe slide valve, from the bend of the spent catalyst carrier line, and from the precipitator standpipes, so that the catalyst systems of the unit can be largely emptied of catalyst without the use of mechanical handling equipment. A "vacuum cleaner" system is also provided to move odd amounts and catalyst spillages to the bag filter separator, with the same compressor serving to pump the carrier air for this line also.

Before putting catalyst into a fluid cracking unit, it is desirable to have process equipment temperatures well above the dew point to prevent formation of catalyst muds and the possible blocking of catalyst flow during operation. An air heater in the carrier air system is used to do this preheating of equipment, hot flue gases being allowed to flow up through the principal piping and vessels along with carrier air. A 20-inch line is provided for transferring catalyst from either storage hopper to the process equipment, utilizing carrier air from the main process air blower of the unit. This catalyst enters the base of the spent catalyst carrier line and is blown into the regenerator by the regular stream of carrier air. The catalyst is moved to the reactor through the regenerated-catalyst standpipe, slide valve, and reactor feed riser by means of a carrier stream of steam. When catalyst is in the system in approximately the desired amount, the equipment is brought up to initial operating temperature by means of the air heater.

Calculations

Fluid-catalyst circulation systems are designed with line sizes and equipment elevations adequate for the desired catalyst flow. The sizing of lines is by direct calculation of the diameter required to give desired design velocities, but the setting of equipment elevations is largely a matter of trial-and-error calculation of catalyst heads and pressure drops.

In Figures 4 and 6 the regenerated catalyst standpipe is longer than the spent catalyst standpipe, for reasons already given. In design of such units the reactor, regenerator, and catalyst stripping section are first worked out from process considerations, and flow quantities are used to calculate flowing catalyst densities in carrier lines. With densities established within an operable range (5 pounds per cubic foot maximum for design), equipment elevations are set up. The inlet end of the spent-catalyst carrier line is established at a convenient elevation above ground level and the reactor set at a trial elevation providing a standpipe length estimated as reasonable. A summation of pressure drops from the fractionator reflux-drum to the inlet of the reactor feed riser gives the pressure required at that point, and the regenerated catalyst standpipe height is calculated to equal this pressure plus the design slide-valve pressure drop, thus setting the height of the regenerator. A summation of pressure drops from the regenerator outlet back to the inlet of the spent-catalyst carrier line gives the pressure required below the spent-catalyst slide valve, and a check of spent-catalyst standpipe head and available slide-valve pressure drop completes the elevation calculations. If the available slide-valve pressure drop is not as desired, the reactor elevation is changed to correct the spent-catalyst standpipe length, and all calculations are repeated, until a satisfactory balance is obtained. With other designs, different pressure levels in reactor and regenerator will result in different relative elevations between these vessels, but the general calculation method is the same.

The volume of the gases which leave the reactor and regenerator with the flowing catalyst, and the volume of aeration gases can be calculated from catalyst and gas densities as follows:

$$\rho_c (1-x) + \rho_g x = \rho_{fc}$$

$$x = \frac{\rho_c - \rho_{fc}}{\rho_c - \rho_g}$$

where

x = volume of gas per cubic foot of flowing catalyst
$1-x$ = true volume of catalyst per cubic foot of flowing catalyst
ρ_c = true density of catalyst, pounds per cubic foot.
ρ_{fc} = density of flowing catalyst, pounds per cubic foot.
ρ_g = density of gas calculated for existing temperature and pressure, pounds per cubic foot.

$$V = \frac{Wx}{\rho_c (1-x)} = \frac{W\left(\frac{\rho_c - \rho_{fc}}{\rho_c - \rho_g}\right)}{\rho_c\left(1 - \frac{\rho_c - \rho_{fc}}{\rho_c - \rho_g}\right)} = \frac{W(\rho_c - \rho_{fc})}{\rho_c (\rho_{fc} - \rho_g)}$$

V = volume of gas leaving vessel, cubic feet per hour
W = true weight of catalyst leaving vessel, pounds per hour

Calculating the density, volume, and weight of gas flowing into a standpipe from reactor or regenerator "dense-phase" catalyst bed, and making similar calculations for the gas required at the bottom of the standpipe, entering the slide valve, allows aeration

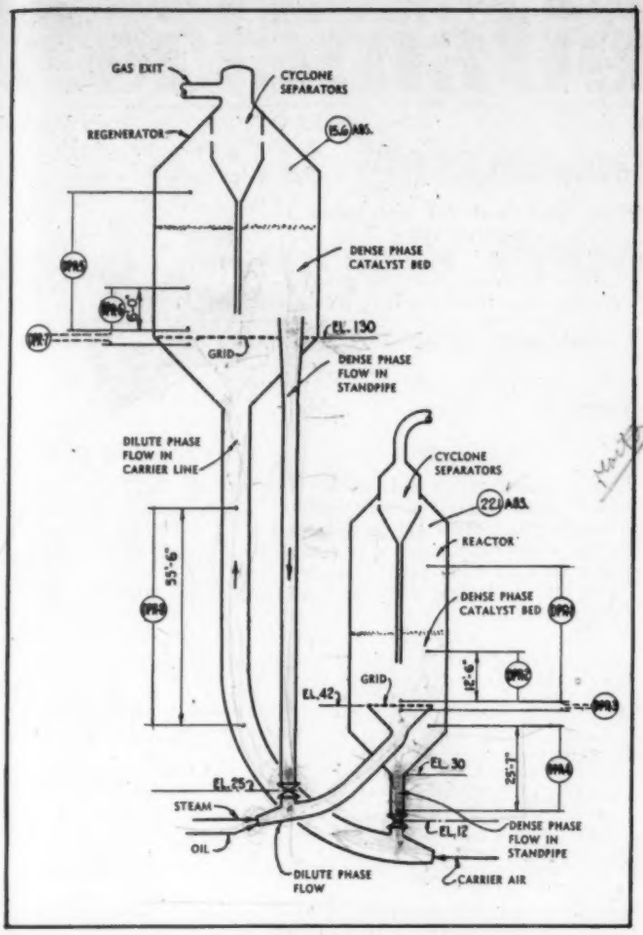

FIGURE 6

gas to be determined by difference. Aeration gas required for a given flowing catalyst density at any standpipe level can be calculated in a similar manner.

Pressure drop across distribution grid orifices and slide-valve openings can be calculated, approximately, from a standard orifice entrance loss formula as follows:

$$c\sqrt{2gH} = \sqrt{V_0^2 - V_1^2}$$

$$H = \frac{V_0^2 - V_1^2}{2g\,(c)^2}$$

$$H = \frac{144\,\Delta p}{\rho_{fc}}$$

$$\Delta p = \frac{\rho_{fc}\,(V_0^2 - V_1^2)}{144\,(2 \times 32.17)c^2} = \frac{0.000108\,\rho_{fc}(V_0^2 - V_1^2)}{c^2}$$

Δp = pressure drop, pounds per square inch
H = head loss, feet
ρ_{fc} = density of flowing stream, pounds per cubic foot
V_0 = velocity through restriction, feet per second
V_1 = upstream velocity, feet per second
g = acceleration due to gravity, 32.17 feet per second per second
c = orifice coefficient, 0.6 minimum to 0.8 maximum

Examples

To illustrate the calculation of fluid catalyst circulation systems, heads and pressure drops will be calculated and compared with actual operating values. The following quantities and operating conditions are taken from the plant operation shown on Figure 4. In the case of a new design they would be established by process considerations for the particular case. Figure 6 shows equipment elevations, reactor and regenerator pressures above catalyst beds, and differential pressure readings from which density of catalyst beds will be calculated. In new equipment design the standpipe densities would be set at about 25 pounds per cubic foot, while the catalyst-bed densities in reactor and regenerator would be set at about 15 to 20 pounds per cubic foot to provide ample bed volumes for reaction and regeneration to occur even at low catalyst densities.

	°API	BPSD	GPH	#/G	#/H	MW	M/H
Fresh oil feed	28.5	11000	19250	7.364	142000	255	555
Heavy cycle oil recycle	23.0	1000	1750	7.627	13350	200	67
Slurry oil recycle	13.0	650	1135	8.155	9250	280	33
Total oil to reactor		12650	22135		164600		655
Steam to reactor inlet riser					1600	18	89

Coke production, pounds per hour 9000
Pressure above regenerator catalyst bed, psia 22.1
Pressure above regenerator catalyst bed, psia 15.6
Reactor bed temperature, °F 890
Regenerator bed temperature, °F 1100
Reactor feed riser temperature, °F 850
Spent-catalyst carrier line temperature, °F 870
Catalyst circulation (by reactor heat balance) = 16. tons/hour
= 1930000 lbs/hour

Catalyst/Oil ratio = 1930000/164600 = 11.7

Carrier air
24800 cubic feet per minute @ 60° F. and 14.7 psia, dry.

Cottrell return air
883 cubic feet per minute @ 60° F. and 14.7 psia, dry.

With blower inlet at 90° F. @ 60 percent relative humidity, moisture = 0.0185 pounds water per pound dry air.

	CFM@60°F.	Mols/Min	MW	#/Min.	CFM@90°F.
Dry air	24800	65.5	28.97	1900	26200
Water	740	1.95	18	35*	780
Humid air	25540	67.45		1935	26980

*(1900 × 0.0185 = 35)

Spent Catalyst Standpipe

Reactor pressure above catalyst bed = 22.1 psia
DPR-1 reading = 78 inches water
DPR-2 reading = 74 inches water
Tap spacing = 12.5 feet

$$\text{Reactor bed density} = \frac{74 \times 0.433 \times 144}{12 \times 12.5} = 30.8\ \#/\text{cf}$$

$$\text{Bed depth} = \frac{78 \times 12.5}{74} = 13.2 \text{ feet}$$

$$\text{Head of catalyst bed} = \frac{13.2 \times 30.8}{144} = 2.82 \text{ psi}$$

Pressure above reactor grid = 24.92 psia
DPR-3 reading = 20 inches water = 0.72 psi
Pressure below reactor grid = 25.64 psia
DPR-4 reading = 124 inches water
Tap spacing = 25.58 feet

$$\text{Average density} = \frac{124 \times 0.433 \times 144}{12 \times 25.58} = 25.2\ \#/\text{cf}$$

$$\text{Catalyst head across stripper} = \frac{25.2 \times 12}{144} = 2.1 \text{ psi}$$

Pressure at stripper inlet = 24.92 psia
Pressure at stripper outlet = 27.02 psia

$$\text{Catalyst head across standpipe} = \frac{25.2 \times 18}{144} = 3.15 \text{ psi}$$

Pressure above spent catalyst slide valve = 30.17 psia
Steam density at bottom of stripper, 950° F.,

$$= \frac{18 \times 520 \times 27.02}{379 \times 1410 \times 14.7} = 0.0322\ \#/\text{cf}$$

Steam leaving stripper with spent catalyst

$$= \frac{1939000\,(130 - 25.2)}{130\,(25.2 - 0.0322)} = 62000 \text{ cf/h @ } 0.0322\ \#/\text{cf}$$
$$= 2000\ \#/\text{h}$$
$$= 111 \text{ mols/h}$$

Steam density above slide valve, 950° F.

$$= \frac{18 \times 520 \times 30.17}{379 \times 1410 \times 14.7} = 0.036\ \#/\text{cf}$$

Steam entering slide valve

$$= \frac{1939000\,(130 - 25.2)}{130\,(25.2 - 0.036)} = 62000 \text{ cf/h @ } 0.036\ \#/\text{cf}$$
$$= 2230\ \#/\text{h}$$
$$= 124 \text{ mols/h}$$

Aeration steam = 230 #/h
= 13 mols/h

[*Continued on next page*]

Regenerated Catalyst Standpipe

Regenerator pressure above catalyst bed = 15.6 psia
DPR-5 reading = 68 inches water
DPR-6 reading = 33 inches water
Tap spacing 6.0 feet

Regenerator bed density = $\frac{33 \times 0.433 \times 144}{12 \times 6.0} = 28.6$ #/cf

Bed depth = $\frac{68 \times 6.0}{33} = 12.4$ feet

Head of catalyst bed = $\frac{12.4 \times 28.6}{144}$ = 2.46 psi

Pressure above regenerator grid =	18.06 psia
DPR-7 reading = 17 inches water =	0.61 psi
Pressure below regenerator grid =	18.67 psia
Pressure above regenerated catalyst standpipe slide valve =	36.7 psia
Pressure above regenerator grid =	18.06 psia
Regenerated catalyst standpipe head =	18.64 psi

Elevation differential = 130 − 25 = 105 feet

Standpipe density = $\frac{18.64 \times 144}{105} = 25.6$ #/cf

Gas density entering standpipe, inert gas, 29 molecular weight, 1100° F.

$= \frac{29 \times 520 \times 18.06}{379 \times 1560 \times 14.7} = 0.0313$ #/cf

Gas entering standpipe with catalyst

$= \frac{1930000\ (130 - 28.6)}{130\ (28.6 - 0.0313)} = 52700$ cf/h @ 0.0313 #/cf

$= 1650$ #/h
$= 57$ mols/h

Steam used for aeration
Gas density entering slide valve, 1080° F., assumed 22.6 molecular weight

$= \frac{22.6 \times 520 \times 36.7}{379 \times 1540 \times 14.7} = 0.0505$ #/cf

Gas entering slide valve with catalyst

$= \frac{1930000\ (130 - 25.6)}{130\ (25.6 - 0.0505)} = 60800$ cf/h @ 0.0505 #/cf

$= 3080$ #/h
$= 136$ mols/h

	CF/H	ρ	#/H	MW	Mols/H
Inerts			1650	29	57
Aeration steam			1430	18	79
Total gas	60800	0.0505	3080	22.6	136

Reactor feed riser, 850° F., average pressure assumed 26 psia

	#/H	MW	Mols/H
Fresh feed	142000	255	555
Heavy cycle oil	13350	200	67
Slurry oil	9250	280	33
Total oil	164600		655
Inlet steam	1600	18	89
Inerts	1650	29	57
Aeration steam	1430	18	79
Total vapor	169280		880

Vapor volume = $\frac{880 \times 10.72 \times 1310}{26} = 476000$ cf/h

	#/H	ρ	CF/H
Vapors	169280		476000
Catalyst	1930000	130	14800
	2099280		490800

Flowing density $\frac{2099280}{490800} = 4.27$ #/cf

Riser head (with 2:1 "slip factor")

$= \frac{4.27 \times 17 \times 2}{144}$ = 1.01 psi

Pressure below reactor grid	25.64 psia
Pressure loss from friction, bends (estimated)	1.0 psi
Pressure below regenerated catalyst slide valve	27.65 psia

Spent catalyst carrier line, 870° F., average pressure assumed 23 psia

	Mols/H	MW	#/H
Dry air	3930	29	114000
Water vapor	117	18	2100
Aeration steam	13	18	230
Total vapor	4060		116330

Vapor volume = $\frac{4060 \times 10.72 \times 1330}{23} = 2520000$ cf/h

	#/H	ρ	CF/H
Vapors	116330		2520000
Catalyst	1930000	130	14800
Coke	9000	45	200
	2055330		2535000

Flowing density = $\frac{2055330}{2535000} = 0.81$ #/cf

Riser head (with 2:1 "slip factor")

$= \frac{0.81 \times 118 \times 2}{144}$ = 1.33 psi

Pressure below regenerator grid	18.67 psia
Pressure loss from friction, bends (estimated)	2.0 psi
Pressure below spent catalyst slide valve	22.0 psia

Summation of Calculated Pressures

	psia Calculated	psia Observed
Above spent catalyst slide valve	30.17	31.7
Below spent catalyst slide valve	22.0	22.7
Above regenerated catalyst slide valve	36.7*	36.7
Below regenerated catalyst slide valve	27.65	28.2
Spent catalyst slide valve pressure drop	8.17	9.0
Regenerated catalyst slide valve pressure drop	9.05	8.5

* Not calculated.

Line Velocities

	Calculated, Feet/second	Desirable range, Feet/second
Reactor feed riser, 40" ID $\frac{490800}{3600 \times 8.7}$ =	15.7†	20 to 30
Spent catalyst carrier line, 72" ID $\frac{2535000}{3600 \times 28.3}$ =	24.9	20 to 30
Spent catalyst standpipe, 36" ID $\frac{62000 + 14800 + 200}{3600 \times 7.01}$ =	3.05	2.5 to 6
Regenerated catalyst standpipe, 36" ID $\frac{60800 + 14800}{3600 \times 7.01}$ =	3.0	2.5 to 6

†The cracking reaction greatly increases oil vapor volume because of the formation of low-molecular-weight products. Reaction begins in the inlet riser and volume increase occurring here will somewhat increase this line velocity.

Slide Valve Areas

The regenerated catalyst standpipe has two 14" square port slide valves, area open = 196 square inches = 1.36 square feet.

Velocity through open valve

$= \frac{75600}{3600 \times 1.36} = 15.5$ feet second

and $\Delta P = \frac{0.000108\ (25.6)\ (15.5^2 - 3.0^2)}{(0.8)^2}$

$= 0.99$ psi

Pressure drop across "throttled" valve

$= 9.05 - 0.99 = 8.06$ psi

Velocity through throttled valve

$8.06 = \frac{0.000108\ (25.6)\ (V_0^2 - 3.0^2)}{.64}$

$V^2 - 9.0 = 1865$
$V^2 = 1874$
$V = 43.2$ feet/second

Area required for throttled valve

$= \frac{75600}{3600 \times 43.6} = .481$ square feet

Valve opening $= \frac{.481 \times 144}{14} = 4.95$ inches

Similar calculations can be made for the other slide valves and for grid orifices.

Reclamation of Waste

At Beaumont Refinery of Magnolia Petroleum Company

J. L. HEATH and PERCY GIBLIN
Refining Division, Magnolia Petroleum Company, Beaumont, Texas

IN PLANT PRACTICE, appreciable quantities of oil are wasted in handling and treating operations. However, this oil does not represent necessarily a complete loss to a refinery equipped with modern facilities for its recovery. In addition to the actual monetary value of the recovered oil which may be used as fuel, such processes protect the refinery from numerous claims for damages that may arise as a result of polluting water streams and adjacent territories with waste products detrimental to animal and plant life. For these reasons, the problem of reducing the quantity of discarded oil from the plant should be kept always at the lowest possible minimum.

Waste products from refinery operations may contain widely different percentages of oil which determines to a considerable extent the selection of a proper method for their economical handling. The water may contain particles of suspended oil, or the oil may contain particles of suspended water, which can be easily separated by mere settling. In other instances the two major constituents may form emulsions of water-in-oil or of oil-in-water type, requiring somewhat elaborate methods for their resolution. The second type of emulsion is much more common around the refineries than the first type, which is prevalent in the oil fields.

Emulsions

Emulsions consist of minute globules of one liquid surrounded by a film of the so-called emulsifying agent and suspended in another liquid. Emulsifying agent prevents the globules from uniting with each other and forming larger particles that would settle out by gravity difference. Sodium soaps produced in

FIGURE 1
Inlet of a typical inter-refinery separator. Feed to the separator consists of slop oil containing comparatively small quantities of water. The oil layer obtained after settling is pumped into the black tank shown on the left.

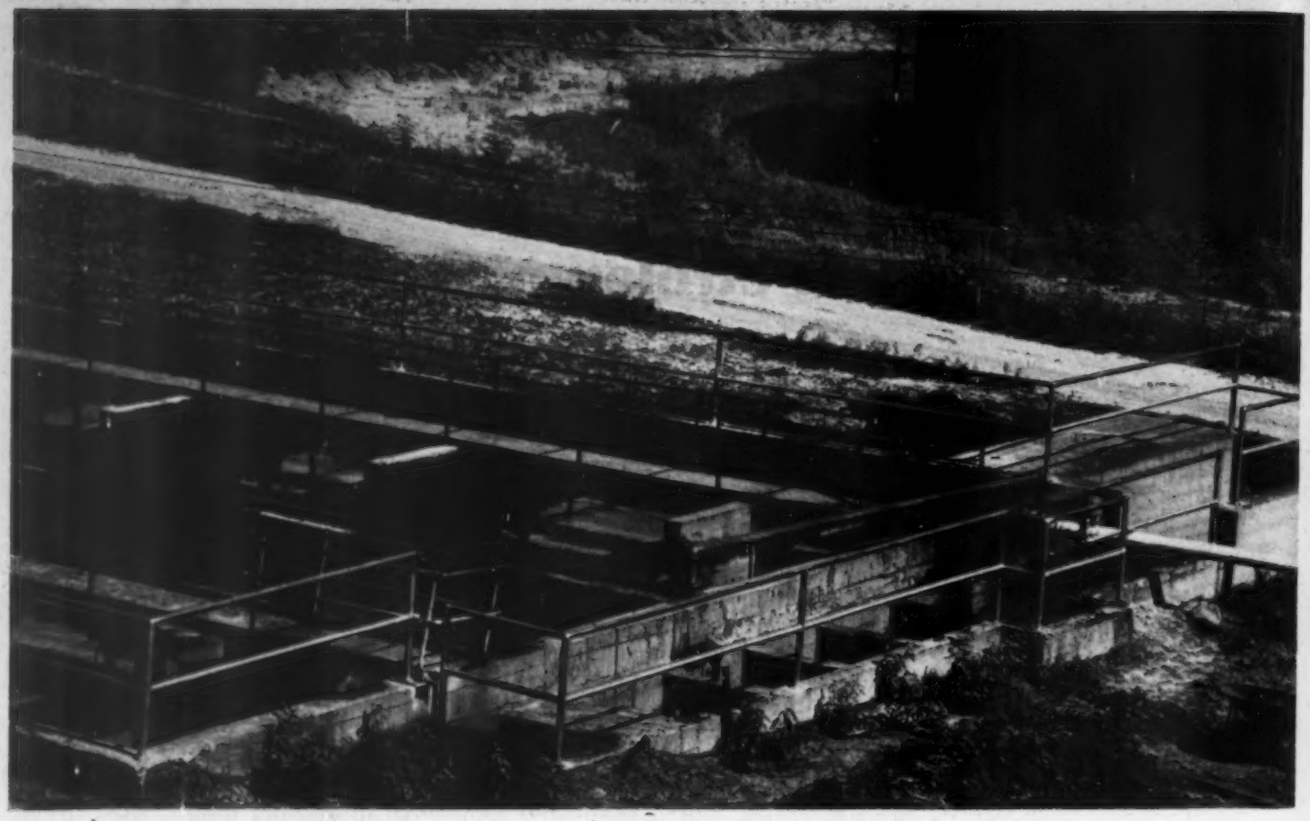

FIGURE 2

Outlet of an inter-refinery separator. Note the system of baffles used for improving separation of water from the emulsion.

FIGURE 3

Inlet of waste-oil settling pond (right). Pond is used for settling water containing relatively small quantities of oil. Water layer obtained from inter-refinery separators passes through this pond before it is discharged from the refinery.

FIGURE 4

Outlet from waste-oil settling pond (left). Note the system of baffles designed for improving separation of water and oil layers.

refining oils with caustic soda are common emulsifying agents responsible for the oil-in-water type of emulsions, while asphalt, clay, lead sulfide or calcium soaps give emulsions of the other type. It may be readily seen that emulsifying agents responsible for one type of emulsion are capable of breaking emulsions of the opposite type.

Waste refinery streams containing small quantities of oil in suspension are normally handled by passing them through settling tanks equipped with suitable traps and level control devices[1]. In some instances the water layer may be filtered through spent clay or small particles of coke in order to improve the separation and assist in breaking emulsions which are

FIGURE 5

Slop-oil emulsion-breaking tanks are the two large tanks to the left and to the right on the picture. Spent caustic is stored in the small tank shown on the foreground.

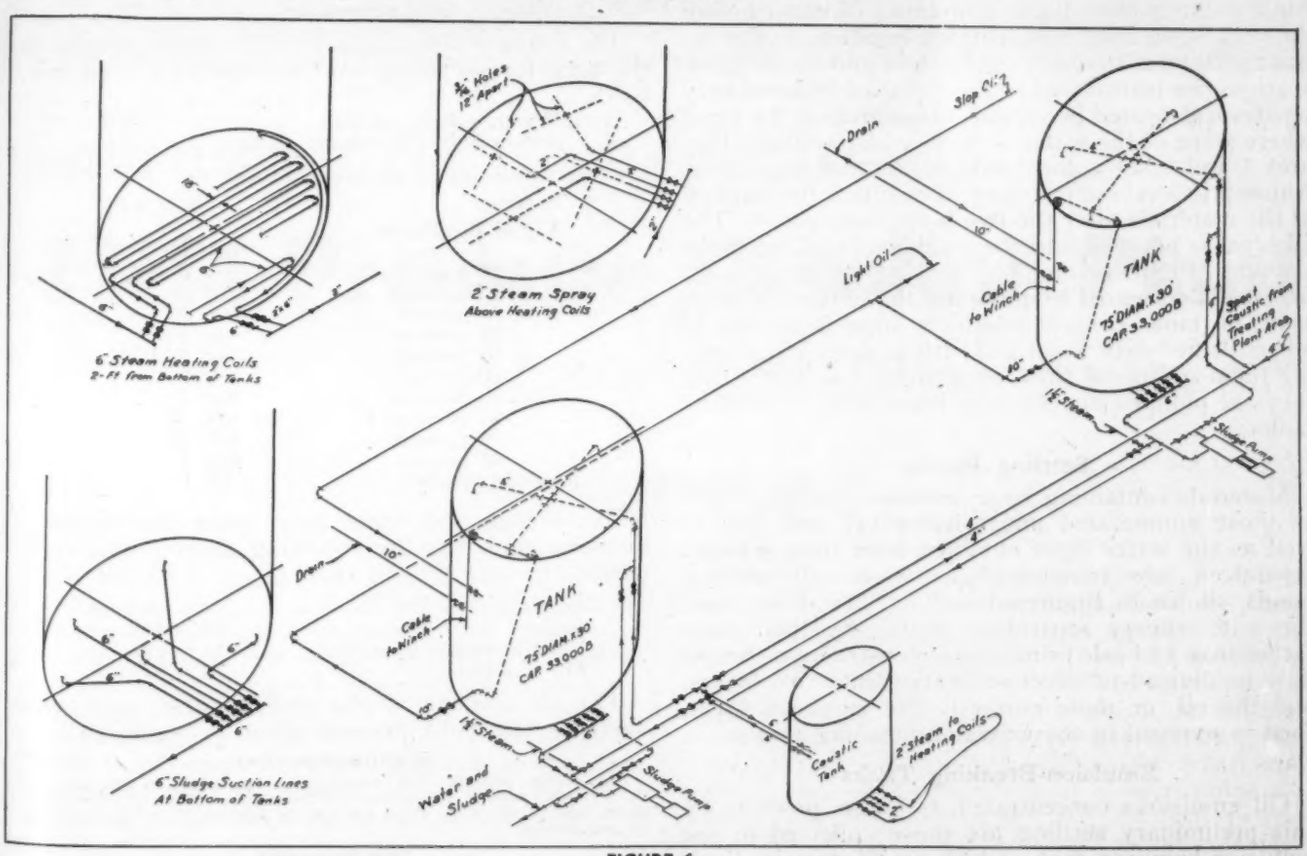

FIGURE 6

Waste-oil system, Magnolia Petroleum Company's Beaumont refinery.

relatively unstable. However, refinery wastes containing large quantities of emulsified oil are not susceptible to such treatment. Some refineries attempt to handle each individual stream separately, depending on its inherent characteristics, but this requires multiplication of equipment and efforts resulting in poor overall economy. These objections were successfully overcome at the Beaumont Refinery of Magnolia Petroleum Company, where the method described below has been in use for a number of years and is now of proven commercial value. It is of interest that the effluent water is of better quality than the water entering the refinery. The emulsion-breaking system is rather unique in that only refinery waste products are used and in that the process appears to lend itself to breaking of all types of emulsions which originate within the refinery.

Sources of Waste Materials

Major sources of waste materials treated at the Beaumont Refinery may be classified as follows:

1. Oil rejected from various treating units or spilled in this refinery area.
2. Bottom settlings and water (BS&W) from crude-oil tanks which are removed daily by pumping.
3. Oil displaced from pipe lines in order to avoid contamination caused by pumping different products through the same conduits as well as materials lost by spillage, leakage or breakage of equipment.
4. Refuses resulting from tank cleaning operations.
5. Oil present in waste-treating reagents or wash waters.

These materials are gathered in various parts of the refinery in an integrated system of sewers and settling tanks or ponds. Some of them are handled in a slightly different way than others in order to remove the maximum feasible quantity of water before they reach the final demulsifying equipment. For instance, materials answering the first and third classifications are transferred to the so-called inter-refinery separators, located in various areas around the plant where some of the water is removed by settling. Figures 1 and 2 show construction of these separators. Automatic level-control devices regulate the outflow of the materials after the two layers are formed. The oil layer is pumped into the emulsion-breaking tanks shown in Figures 5 and 1.

Materials secured by pumping the bottom layer of crude-oil tanks contain relatively large quantities of oil emulsified with water and little is gained by allowing them additional time for settling. For this reason they are pumped directly into the emulsion-breaking tanks.

Settling Ponds

Materials containing large volumes of water, such as those enumerated under items (4) and (5), as well as the water layer obtained from inter-refinery separators, are transferred to waste-oil-settling ponds, shown in Figures 3 and 4. They differ from the inter-refinery separators mainly by their sizes rather than by basic principles of construction. Ample time is allowed to effect separation into two layers, and the oil, or more correctly the emulsion layer, then is pumped in the emulsion-breaking tanks.

Emulsion-Breaking Tanks

Oil emulsions concentrated as far as possible by this preliminary settling are those collected in the emulsion-breaking tanks which are 75 feet in diameter and 30 feet high. Details of their construction are shown in Figure 6. It may be noted that these tanks are equipped with flat steam coils for heating and have a centrally located spider in the roof for introducing spent refinery caustic and similar materials which act as emulsion-breaking chemicals. The process is operated as follows:

The emulsion-breaking tank of 33,000 barrels total capacity, which contains 10,000 barrels of material left from the previous cycle is charged with 10,000 additional barrels of emulsion obtained from the sources already described. This emulsion contains normally about 25 percent oil and 75 percent water. The mixture is heated to about 120-150° F. One thousand barrels of boiler blowdown containing 3,000-4,000 ppm solids, are sprayed into the tank from the spider. This is followed by another spray of 100 barrels of spent caustic. The spent caustic is an emulsion by itself as it contains about 22 percent oil.

The mixture is allowed to settle for 24 hours, using a 48-hour operating cycle for each tank. The oil layer at the top and the water and sludge layers at the bottom are pumped out, leaving about 10,000 barrels of unbroken emulsion for the next cycle.

The "clean" oil layer recovered from this operation has the following average characteristics:

ASTM distillation	
I.B.P.	192° F.
10 percent	353° F.
20 percent	413° F.
30 percent	455° F.
40 percent	494° F.
50 percent	536° F.
60 percent	578° F.
70 percent	744° F.
80 precent	683° F.
81 percent	cracking
Gravity	30.5° API
B. S., volume percent	0.1
Water, volume percent	72.9

The intermediate emulsion layer which remains in the tank for recycling has the following characteristics:

Water, volume percent	72.9
Recoverable Oil, volume percent	22.0

Characteristics of recoverable oil:

Gravity	38.1° API
ASTM distillation	
5 percent	124° F.
10 percent	180° F.
20 percent	310° F.
30 percent	364° F.
40 percent	406° F.
50 percent	438° F.
60 percent	466° F.
70 percent	492° F.
80 percent	520° F.
90 percent	550° F.
97 percent	630° F.
Recovery, percent	97° F.

The sludge and water layer from the emulsion breaking tank has the following approximate composition:

Solids	1 — 4% wt.
Water	96 — 99% wt.
Volatile matter in solids	10 — 40% wt.
pH of water	7.8 — 10.5

The oil content of the sludge-water layer thus averages about 0.2 percent which is negligible.

ACKNOWLEDGMENT

The writers express their appreciation to Messrs. J. W. Newton, W. W. Leach and R. R. Jackson for permission to publish this manuscript, and to Mr. J. A. Oslin for helpful suggestions in the course of its preparation.

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UNITED GAS PIPE LINE COMPANY'S CARTHAGE

Field-Pressure Gasoline Plant

PROCESSING 100 MILLION FEET PER DAY

C. C. PRYOR, Associate Editor

THE FIRST large gasoline plant in the Carthage field, Panola County, Texas, owned and operated by United Gas Pipe Line Company is now on stream with capacity for processing 100,000,000 standard cubic feet of gas and producing approximately 170,000 gallons of liquid products daily. An additional 50,000,000 standard cubic feet of gas may be passed through the system of separators, and heaters in the plant and supplies the company's low-pressure (450 psig) gas pipe line. This gas is not processed for its gasoline content.

A high-pressure dehydration unit, capable of handling 100,000,000 cubic feet per day of residue gas from the absorbers (operated at 1040 psig), also is included in the plant. Dehydrated gas from the plant supplies the company's recently completed 24-inch pipe line to Monroe, Louisiana, from the field at a pressure of 1000 psig. The condensate fractionating unit is similar to that of refinery design, permitting simultaneous production of several specification products from the single unit. Designed for maximum flexibility, the plant can produce a wide range of products; propane, isobutane, and liquified petroleum gases, natural gasoline, motor gasoline, naphtha, kerosine, and residue.

The plant and pipe lines operate on field pressure,

and with the exception of one small recompressor for flashed gases, compressors are not used.

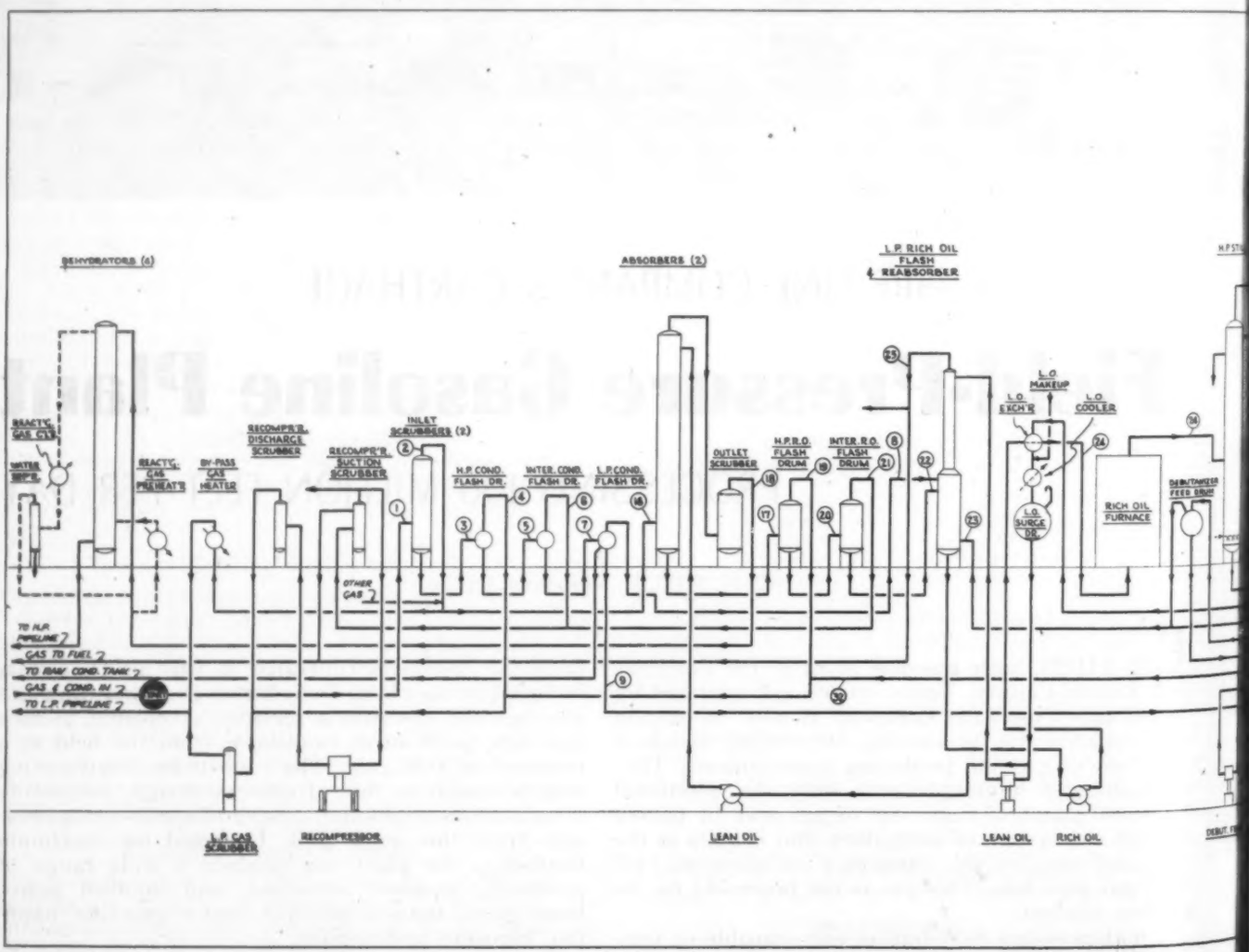
Condensate gas is brought to the plant through a 20-inch line from the field gathering system consisting of 103 miles of pipe, ranging in size from 6 to 20 inches and delivered to four inlet scrubbers 4 x 20 feet, at 1050 psig and 85° F. (Figure 1.) From the scrubbers the gas stream is divided so that 50,000,000 cubic feet daily may be diverted through a bypass heater where steam raises the temperature from 85° F. to 105° F. to prevent hydrate formation, then to the low-pressure pipe line. The remainder of the gas flows to the two 5 x 48-foot high-pressure absorbers (100,000,000 cubic feet daily) where it is contacted by counter-flowing absorption oil (463 g.p.m., 0.815 sp. gr., straight-run kerosine distillate) on 20 trays, which have 79 bubble caps per tray. Absorber residue gas flows through an outlet scrubber to the dehydration unit.

Condensate Processing

Condensate from the inlet scrubbers is flashed in three stages at 500, 200, and 80 psig in the high-, intermediate-, and low-pressure condensate flash drums, respectively. Overhead gas from the high-pressure flash drum, 5 x 20 feet, flows through a scrubber to the low-pressure gas line as does gas from the intermediate flash drum, 5 x 10 feet, after it passes through a suction scrubber for recompressing from 190 psig and 90° F. to 460 psig and 170° F. and then through the scrubber to the low-pressure gas line. The gas from the low-pressure flash drum, 10 x 40 feet, flows to the low-pressure rich-oil reabsorber at the top of the lower section.

The stream of raw condensate, after three stages of flashing, flowing at 70° F. and 80 psig from the low-pressure condensate flash drum, is divided into two streams; one stream flows to the hot-condensate flash tower, 3 x 31.5 feet—12 trays, 27 bubble caps per tray, onto the top tray, and the second stream enters the tower at the tenth tray after passing through a steam-heated preheater, where its temperature is raised to 180° F.

Rich gases overhead from the flash tower flow to the reabsorber entering below the trays in the lower section. Depropanized condensate at 310° F. and 60 psig flow from the hot condensate flash tower reboiler, which is heated by recirculated bottoms from the condensate fractionator, at a rate of 87.4 gallons per minute onto the fourth tray of the fractionator tower, which is 5.52 x 55.5 feet with 20 trays and 103 bubble caps per tray. Hot lean oil also is pumped into this stream from the bottom of the low-pressure still. A portion of the reflux stream to the low-pressure still (3.1 gallons per minute) enters the fractionator on the fourteenth tray. The overhead product of the fractionator, motor gasoline, is refluxed at a rate of 70.5 gallons per minute. Of the total liquid produced overhead, 112 gallons per minute, 41.5 gallons per minute goes to storage. The bottoms of the condensate fractionator are recircu-



Process flow diagram of United Gas Pipe Line

lated through the heat exchanger and then through a condensate furnace. The furnace, a radiant convection type with 36 tubes, 6 inches in diameter and 21 feet long, delivers 10,000,000 Btu per hour. It has a radiant rate of 7640 Btu per square foot per hour and uses 12,620 cubic feet per hour of fuel gas. Bottoms from the fractionator, 290 gallons per minute, enter the furnace at 500° F. and leave at 550° F. with 50 percent vaporization of the charge, which enters on the first tray of the fractionator.

The condensate fractionator serves a three-section side-stream steam stripper, 2.5 x 36.5 feet, section having four trays—18 bubble caps per tray—producing naphtha, kerosine, and residue. The naphtha stripper cut is made from either the fifteenth or seventeeth tray of the fractionator and returns vapor to the eighteenth. This stripper produces 39.5 gallons per minute of naphtha, which is pumped through a cooler to storage. A cut from either the ninth or eleventh tray is made for the kerosine stripper and vapor is returned to the twelfth tray. Kerosine at the rate of 8.22 gallons per minute is pumped through coolers to storage. The residue stripper cut is made on the first bottom tray of the fractionator and the vapor returned to the third tray. Residue, 1.4 gallons per minute, is pumped through the cooler to storage.

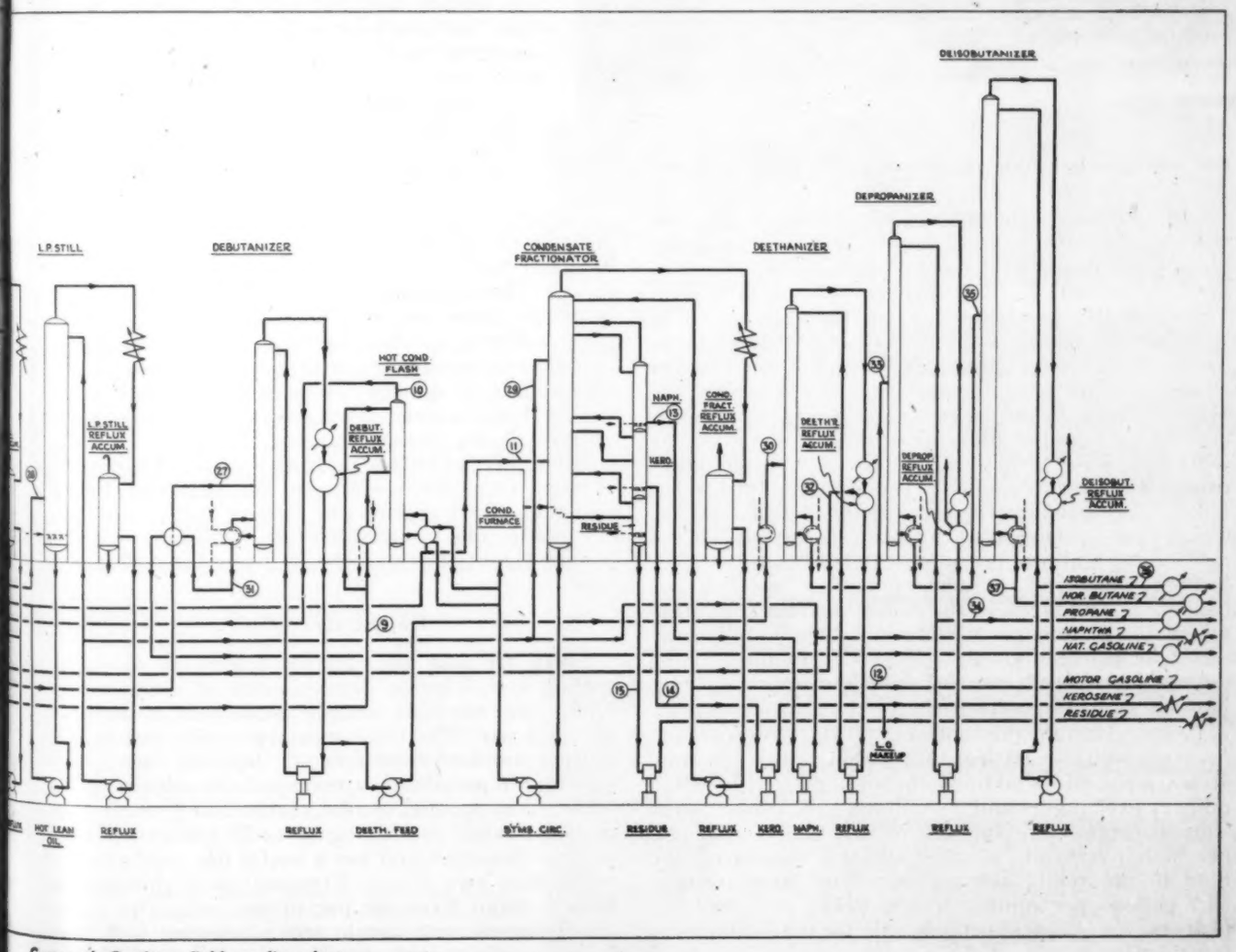
The naphtha stripper is operated at 16 psig and 260° F. on 415 pounds per hour of 25-pound steam; the kerosine stripper at 17 psig and 374° F. on 115 pounds per hour of 25-pound steam, and the residue stripper at 18 psig and 461° F. on 550 pounds per hour of 25-pound steam.

Rich Oil Processing

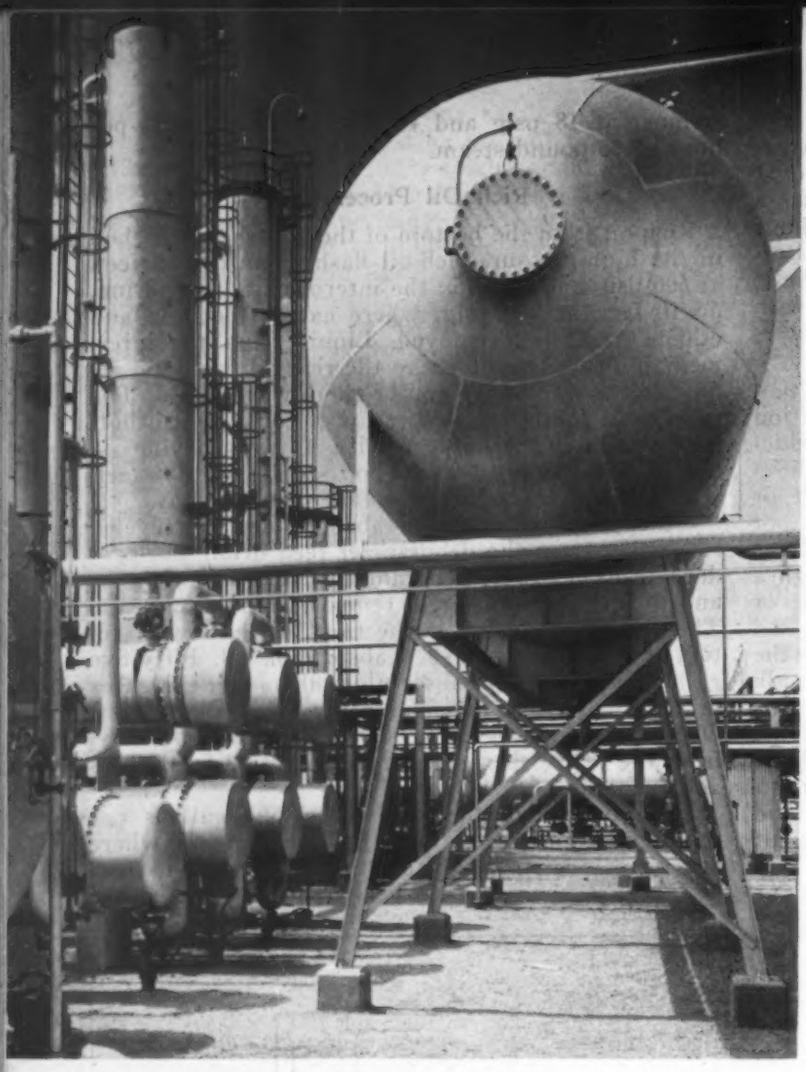
Rich oil from the bottom of the absorber is flashed in the high-pressure rich-oil flash drum, 5 x 10 feet, at 500 psig and again in the intermediate flash drum, 5 x 10 feet, at 200 psig, where excess methane and ethane gases are removed. Liquid from the intermediate drum then flows to the rich oil flash and reabsorber tower at the top of the lower section. The rich oil flash and reabsorber tower consists of a bottom section 5 x 16.75 feet with 4 trays having 48 bubble caps per tray, and a top section 2.5 x 22 feet, which has 10 trays and 18 bubble caps per tray.

Other gases fed into the reabsorber are the recycled rich gases from the debutanizer feed drum, hot-condensate flash tower, debutanizer reflux accumulator, and the high-pressure still reflux accumulator drum. These gases enter below the trays of the lower section to contact the flashed absorption oil. Fifty-five gallons per minute of absorption oil contact the gases in the reabsorber for final control of butane loss. Overhead residue gas, 1,500,000 cubic feet per day, from the reabsorber is used as plant fuel for boilers and furnaces.

The bottoms of the reabsorber, 595 gallons per minute, are pumped through heat exchangers, where 6 percent is vaporized by heating the stream to 340° F. These exchangers are heated by hot lean oil at



Company's Carthage field gasoline plant.



Feed drums, heat exchangers, and still towers in the condensate section.

405° F. pumped from the bottom of the low-pressure still then charged to the rich-oil furnace, which increases the vaporization to 19.5 percent and the temperature to 450° F. This furnace has a heat load of 17,400,000 Btu per hour and is a radiant-convection type furnace having 36 6-inch I.P.S. diameter tubes 29 feet long. It has a radiant rate of 9,620 Btu per square foot per hour. Hourly fuel gas required is 18,875 cubic feet. From the rich-oil furnace, the bottoms are charged to the high-pressure still, 6.5 x 49 feet with 20 trays—146 bubble caps per tray, on the tenth tray. All hydrocarbons, with the exception of the 325 I.B.P. and heavier, are steam stripped from the oil, which then is charged to the low-pressure still at the rate of 521 gallons per minute. Overhead from the high-pressure still, 412,000 cubic feet per hour, is condensed and flows to the reflux accumulator, from which 146 gallons per minute is pumped back as reflux and 44.6 gallons per minute is pumped to the debutanizer feed drum. Gas from the reflux accumulator flows to the reabsorber.

Lean oil from the bottom of the 20-tray low-pressure still, 6 x 50 feet, 121 bubble caps per tray, steam stripped is pumped through the lean oil-rich oil heat exchangers and a shell-and-tube cooler to the lean-oil surge tank. Naphtha vapors, 61,400 cubic feet per hour overhead from the still are condensed and flow to the reflux accumulator. The pump returns 54.7 gallons per minute to the tower as reflux and charges 3.1 gallons per minute to the condensate fractionator.

Fractionation

The 30-tray debutanizer, 4 x 45 feet, 8 bubble caps per tray, operated at a bottom temperature of 295° F. (steam-heated reboiler) and 170 psig, produces debutanized natural gasoline at the rate of 18.7 gallons per minute which flows through the bottoms-feed exchanger, then through coolers to storage. Butanes and lighter overhead from the debutanizer are condensed and flow to the reflux accumulator drum at a rate of 77.7 gallons per minute. Of this volume, 51.8 gallons per minute are pumped back to the tower as reflux and 25.9 gallons per minute are heated in a steam exchanger, which vaporizes 7 percent, and charged to the deethanizer at approximately 215° F.

Bottoms from the deethanizer, principally butanes and propanes, are charged to the depropanizer (33-Table 1). The deethanizer tower is 2.5 x 52.6 feet and has 30 trays with 18 bubble caps per tray. Ethane is removed in the overhead from the deethanizer, which is condensed and passed to the reflux accumulator at 475 psig and 90° F. The ethane is vented to the low-pressure pipe line from the accumulator at a rate of 172,500 cubic feet per hour and 21 gallons per minute of liquid is pumped back as reflux.

Propane vapor is taken overhead from the 2.5 x 68.6-foot depropanizer, which has 40 trays —18 bubble caps per tray, at a rate of 88,300 cubic feet per hour at 122° F. The vapors are condensed and collected in the reflux accumulator drum at 230 psig and 117° F. Liquid propane, 40.4 gallons per minute, from the accumulator drum, 31.6 gallons per minute pumped back to the tower as reflux, and the remainder, 9.8 gallons per minute, is pumped through a cooler to storage.

Butanes from the bottom of the depropanizer are charged to the deisobutanizer (35-Table 1) at 240 psig and 215° F. for separation into isobutane and butane. The isobutane is taken overhead at the rate of 144,000 cubic feet per hour through a condenser to the reflux accumulator at 125° F. and 90 psig. The 77.5 gallons per minute of liquid isobutane from the accumulator is split in two streams, 7.17 gallons per minute being returned as reflux to the tower and 5.8 gallons per minute pumped through a cooler to storage. Normal butane, bottom product of the tower, is taken from the reboiler at 100 psig and 150° F. at a rate of 8.4 gallons per minute, cooled and sent to storage. The tower is 3.5 feet in diameter and 100 feet in height. Each of the 60 trays have 37 bubble caps.

Dehydration Unit

There are four dehydration towers in the dehydrating unit, 5 feet in diameter and 50 feet in height. Each tower contains three separate beds of desiccant of equal size. The total capacity of the unit is 100,000,000 standard cubic feet per day with two towers operated in parallel on stream and the remaining two operated in parallel on the regeneration stream. Activated bauxite containing 60 to 70 percent Al_2O_3 is used as desiccant and has a useful life expectancy of more than two years. Pressure drop through the beds is about 8 pounds per square inch. The towers are designed with ample space between beds with access openings in the tower for replacement of desic-

cant, and for redistribution of the gas stream to prevent channeling.

Gas from the absorber outlet scrubber is saturated with water vapor at the scrubber temperature of 90° F., after being in contact with condensate and water, and has a water dewpoint of 90° F. From the scrubber, the gas flows into the top of the two towers on dehydration stream and the water vapor is adsorbed in the desiccant. Dewpoint of the gas is lowered to less than 30° F. by the dehydrators. The dehydrated gas stream flows directly into the 24-inch transmission line from the Carthage field to Sterlington, 142 miles distant. The line operates on the discharge pressure of the dehydrators of about 1000 psig.

Each pair of towers are on the dehydration stream for a 20-hour period and on the regeneration cycle for 12 hours. The regeneration cycle begins without depressuring the system. A part of the main gas stream ahead of the dehydration unit (about 15 percent) is heated in a tubular heater to 375° F. by 250 psig steam. Flowing from bottom to top in the towers, the hot gas gradually raises the temperature of the beds to release the adsorbed water.

At the higher temperature, the regenerating gas has a high saturation capacity and flows from the towers to the gas cooler, where the water is condensed and drawn off the bottom of the separator. This gas then may be returned to the main gas stream entering the towers for dehydration, or to the dry gas stream to the pipe line, depending on the dewpoint that is required of the gas to the pipe line.

In regenerating the beds, the time required depends on the volume of regenerating gas and its temperature. The temperature at the top of the bed is used as the control point and when it reaches 325 to 350° F. regeneration is completed, the heater is by-passed and cool gas passed through the beds until they return to normal temperature.

The regenerating gas flowing upward through the beds removes traces of absorption oil deposited by the gas on the downflow dehydrating cycle. Downflow of the large volumes of gas during dehydration creates less disturbance of the bed due to pressure drop, because the desiccant particles have a reduced tendency to be shifted by the downward pressure of the gas. As a result, the particles do not grind against each other and are not entrained in the gas stream leaving the towers.

Major Equipment

The plant requires 47,115 pounds per hour of 250-psig dry saturated steam, 9,255 pounds per hour of 100-psig steam, and 11,895 pounds per hour of 25-pound steam, which is supplied from a steam plant of two water-tube boilers, either of which is capable of delivering the required steam while the other serves as an auxiliary. Each boiler requires 55,700 standard cubic feet per hour of fuel gas and delivers steam at 406° F. Total effective heating surface is 8,278 square feet, water-cooled boiler surface is 204 square feet, heat consumption is 95,000 Btu per square foot per hour, furnace heat release is 28,600 Btu per square foot and overall efficiency is 77.5 percent. Each boiler has 6 burners.

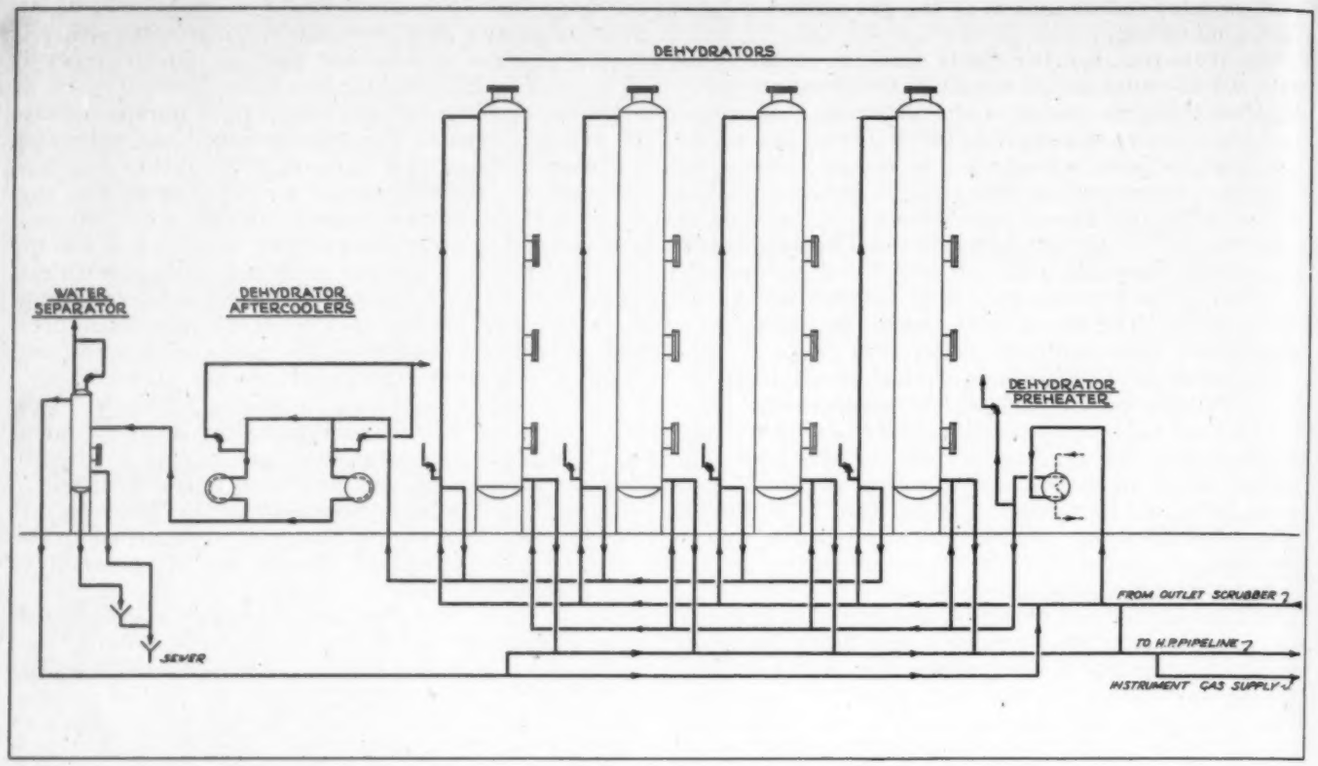
Steam turbines driving centrifugal pumps operate on 250-psig steam. The high-pressure lean oil pump turbines exhaust at a vacuum of 27 inches Hg, and the exhaust steam returned to the boilers. The turbines for the rich-oil pumps operate with 100 psig back pressure with the exhaust steam used for the greater portion of the reciprocating pumps, reboilers, and process steam. Turbines for the hot-lean-oil and cooling-water pumps operate at 25 psig back pressure and the exhaust steam is used for steam-heated tubular heat exchangers and process steam.

Other pumps in the plant operate at about 5 psig back pressure with a portion of this exhaust going to jacket heaters on the inlet gas header to the absorbers when required; otherwise it is exhausted to atmosphere. The condensate from the 250-psig and 100-psig steam-heated exchangers is returned to the boilers and the excess 25-psig steam returned to the deaerating heater.

The cooling tower for the plant is an induced draft type with a coil shed for atmospheric cooling and condensing sections. Water is cooled from 98 to 80° F. with a wet-bulb temperature of 74° F. The makeup water is supplied from two wells on the property pumped by 200-gallons-per-minute motor-driven pumps.

The structure is 144 feet long, 30.5 feet wide and 40.75 feet high. It accommodates 18-6 x 24 foot bays of atmospheric sections with the coil shed 11 feet high, 24 feet wide, and 144 feet long. There are six cells in line, each 30.5 x 24 feet. Each cell has a four-bladed

One of two outside control panels for controlling instruments in the plant. Connecting lines are carried in vertical banks rather than horizontal layers to simplify installation and maintenance. Note orifice connection at uppper right.



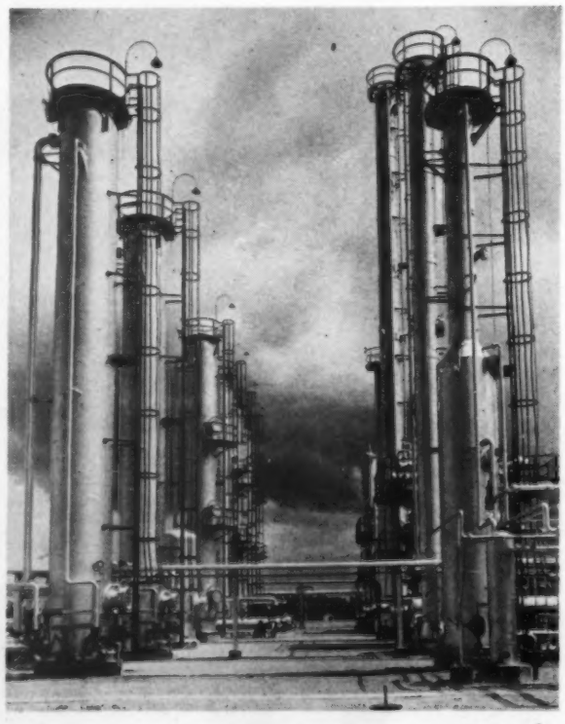
Flow diagram of the plant dehydration unit. The four columns have a capacity of 100 million cubic feet daily of gas. When two are on stream the remaining two are being regenerated.

fan driven at 200 revolutions per minute by a 440-volt explosion-proof electric motor rated at 25 horsepower.

Maximum capacity to the atmospheric sections is 4,300 gallons per minute and to the plant 2,700, a total of 7000 gallons per minute. Two pumps supply circulation; one pumping cold water through shell-and-tube units and over the cooling tower, and the other circulating warm water from atmospheric sections over the tower. Cooled water in the tower is collected at the base of the cooling section and flows through a trough from which the requirements of the atmospheric sections flow through distributing pipes to spray heads under manual control. After passing over the atmospheric sections the water flows into the warm section of the cooling-tower basin. From the basin, the warm and cold water enter the cooling-tower pump pit, which is divided into warm and cold sections from which the circulating pumps take suction.

The boilers, direct-fired oil heaters, and the recompressor gas-engine drive obtain their fuel gas from the reabsorber residue and a portion of the vent gases from the intermediate-pressure condensate and rich-oil flash drums. The gas has an average heating value of about 1220 Btu per standard cubic foot and a specific gravity of 0.759. Of the total hourly consumption of fuel gas, 88,505 standard cubic feet, the reabsorber supplies 70 percent.

Electric power for the plant is supplied by the local power company and is used for lighting and miscellaneous services. Total requirement is 425 KVA.



The two columns in the left foreground are the absorbers, behind them are the dehydrator columns. Reabsorber and fractionating columns are on the right.

Acknowledgement

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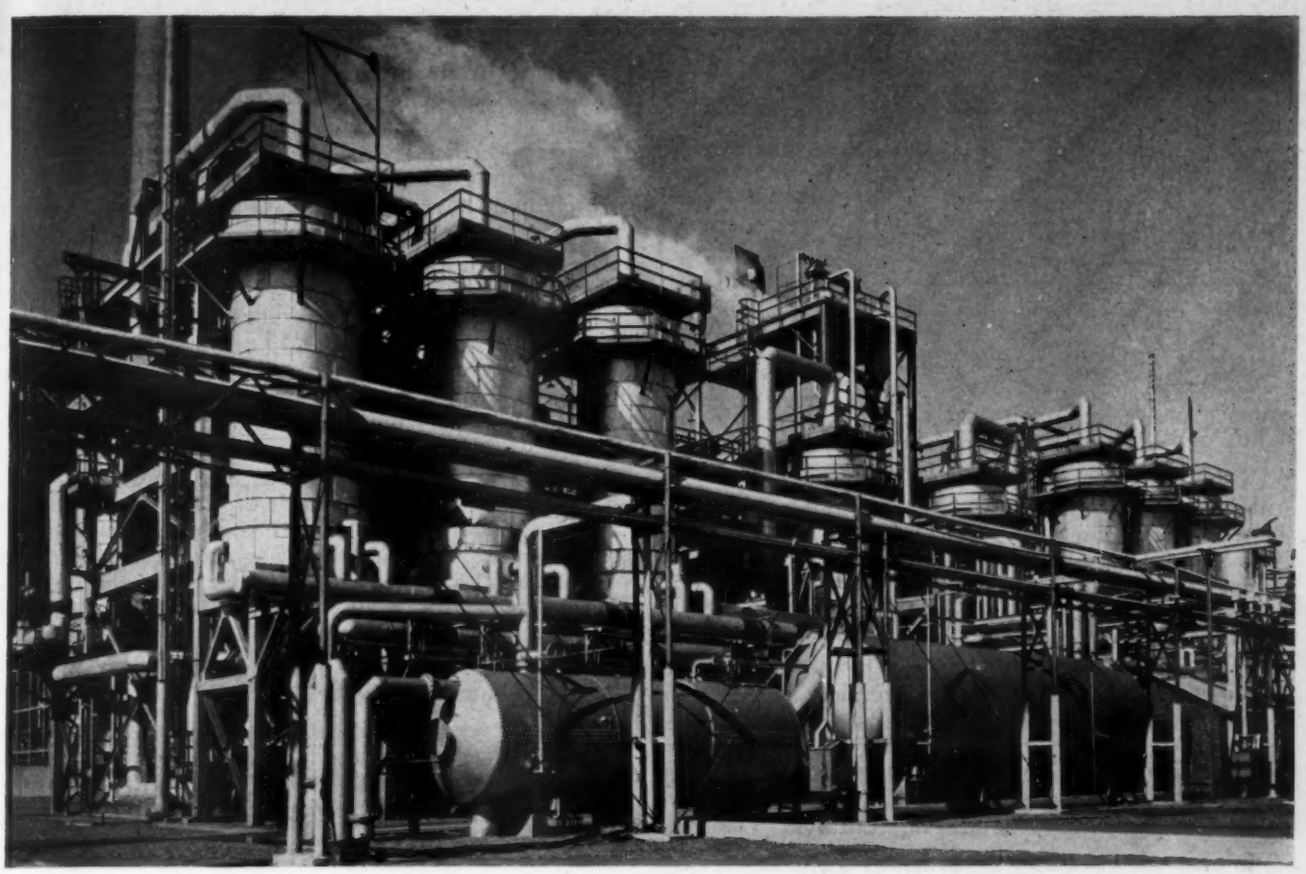

Improved Houdry Reactor Design

R. C. LASSIAT, Houdry Processs Corporation
C. H. THAYER, Sun Oil Company

A MAJOR improvement in the Houdry Catalytic Cracking Process, resulting in both lower operating costs and increased gasoline production, has been effected by re-designing the reactor tubes and changing the physical composition of the catalytic mass.

The new design, developed by engineers of Houdry Process Corporation and Sun Oil Company, is adaptable to all standard Houdry reactors and its advantages have been fully demonstrated by a year's operating experience with a 12-case cracking plant at Sun Oil Company's Toledo, Ohio, refinery. A similar 12-case, 20,000-barrel plant at the company's Marcus Hook, Pennsylvania, refinery, has also been equipped with tubes of the new design. In addition, Sun Oil Company recently has completed conversion of a third 12-case plant with tubes containing further improvements over those installed in the first two units.

Cost of Changing Catalyst Sharply Cut

The cost normally involved in changing catalyst—an item of importance in the operation of Houdry fixed-bed plants—has been markedly reduced by the improved design. Moreover, the new tubes are sturdier and will give longer life, and metal temperatures are lower than heretofore experienced under similar operating conditions.

With the new design, increased production of gasoline results from higher on-stream efficiencies coupled with more even distribution of oil vapor and regeneration air through the catalyst and the presence of a greater supply of heat within the catalytic mass for the cracking reaction. The increased heat supply is provided by a granular chemically-inert material mixed with the catalyst, which has the effect of augmenting the heat capacity of the catalytic mass, as well as other advantages which are described in greater detail in this article.

Conversion of Reactors Is Simple

All distributing orifices and shields under the new design are contained on tubes which are easily removable from the top of the reactor for inspection when changing catalyst. Thus complete cleaning of all distributing elements is possible at no loss of

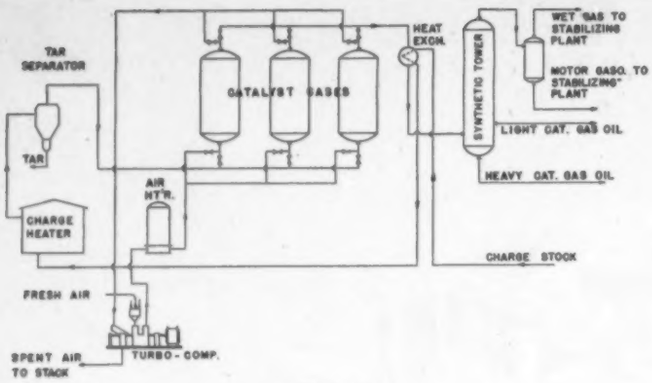

FIGURE 1
Flow diagram of Houdry Catalytic Cracking Unit.

time on every catalyst change, resulting in improved distribution of air and hydrocarbon vapors. Existing Houdry reactors can be converted to the new type simply by changing tubes, jacket liner and top tube sheet. Since these items ordinarily have to be renewed after five or six years' operation, no additional expenditure is involved beyond the normal cost of replacement by making the conversion at this time. Since the new tube design results in an increased volume of catalyst space, the addition of inert material does not change the active catalyst capacity.

Practice in Changing Catalyst

In Sun Oil Company's operations with the previous design of reactors, catalyst life when producing motor gasoline has averaged about 18 months. Starting with a new plant, the usual practice when making the first catalyst change has been to "shake" out the catalyst. This is accomplished by removing the top head, top catalyst tube sheet and the regeneration tubes which can be unscrewed from the top. Vibrators are then placed on the cooling and collector tubes which remain in place, and the catalyst falls out of the bottom of the reactor through the holes provided by removal of the regeneration tubes. About 18 months later, when the second catalyst change is made, all of the tubes are removed from the cases for cleaning and repair. This work consists mostly of removing small particles of catalyst, iron oxide, etc., which accumulate between distributing tubes and orifice-protecting shields during operation. straightening out tubes and repairing threaded parts which may have been impaired during dismantling.

When making catalyst changes it is general practice to shutdown half of the reactors in a unit and, by changing full throughput through the remaining half, the overall gasoline production during the catalyst change period is about 75 percent of normal. The shutdown time for one set of six reactors of the previous design for the first catalyst change has averaged 20 days with a minimum labor requirement of 4000 man-hours per case. The second catalyst change, when all of the tubes are removed, usually has required a shutdown time of 6 to 7 weeks, during which 12,000 man-hours are required per case. A few months ago, by efficient scheduling of all operations, this job was done on a six-case plant of the previous design in 29 days from off-stream to on-stream.

Shutdown Time Materially Reduced

Catalyst changes, whether it be the first, second or any subsequent change, with the new type of tube require only 7 to 10 days from stream to stream and 1200 to 1500 man-hours per case. Inasmuch as all of the tubes which contain distributing orifices are easily removable from the top of the reactors, distribution of oil vapors and air through the catalytic mass can be maintained almost perfectly without the necessity of removing any of the salt tubes. An improved design has been made for the periphery of the reactors to eliminate any tendency for undue heating of the liner, border tubes or the shell of the reactors.

As will be seen in the following descriptive section, the interlocking features of the fins have been eliminated to facilitate removal of catalyst and the new fins are extremely sturdy and heavily welded to the cooling tubes. Due to the heavy section of the fins,

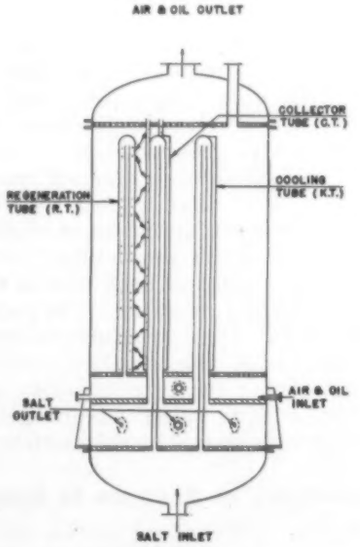

FIGURE 2
Vertical cross section of Houdry catalytic case (schematic).

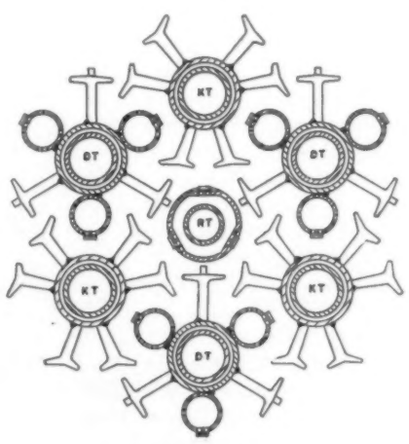

FIGURE 2-A
Typical tube pattern, Houdry catalytic case.

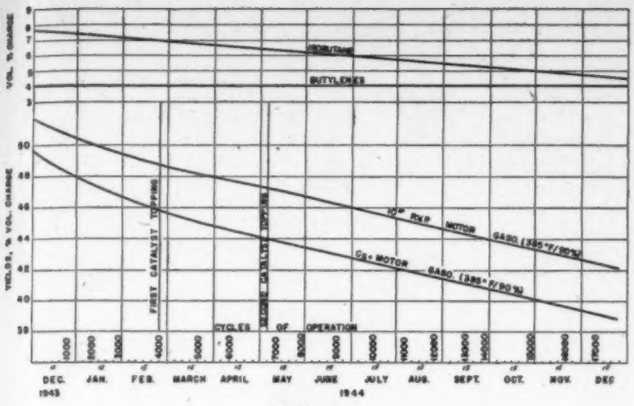

FIGURE 3

Catalytic gasoline and butane yield, Plant 11-4, December 1943-44, inclusive.

the additional welding and the reduced surface, the metal temperatures are considerably reduced. The change in design, as shown by results currently obtained in Sun Oil Company's operations, has resulted in increased production of gasoline through maintenance of the distributing elements in better condition and higher on-stream efficiencies.

Design Factors of Houdry Reactors

The fixed-bed principle, as utilized in the Houdry process, is familiar to the oil industry and has been described at length.

Briefly, reactors are usually arranged in groups of three, oil vapors being circulated through one of the reactors, while air is passed through another to burn off the coke deposited on the catalyst by a former on-stream period. The third reactor, at the same time, undergoes the processes of oil purge, air purge and valve changing.

Necessary piping and motor-operated valves insure the continuity of oil and air flow through the system. The valves are operated by an automatic cycle timer and proper electric interlocks preclude mixing of streams.

A molten salt circulated through all reactors removes the excess heat of combustion of the coke and controls the general temperature level.

Figure 1 shows a simplified flow diagram of a typical Houdry Fixed-Bed Unit.

Reactor Design

The design of the fixed bed reactors is governed by the following factors:

1. Rate of heat removal of the order of 10,000 Btu per hour per cubic foot of catalyst space.
2. Temperature control during the cracking period.
3. Limitation of maximum temperature, during regeneration period, below values detrimental to catalyst life.
4. Catalyst bed depth and accurate distribution of vapors and air.
5. Mechanical considerations of assembly, thermal expansion, catalyst loading and unloading, etc.

Figures 2 and 2A show the general arrangement in vertical and horizontal sections of a type of reactor widely used.

Several years of commercial operation have shown that insofar as temperature control, yields and catalyst life are concerned, the design has been satisfactory. Results predicted in laboratory evaluations have been maintained over long periods of time as illustrated by Figure 3 which shows a typical plant-performance diagram over a period of 12 months.

In this plant a heavy distillate obtained mostly from East Texas crude was processed for the production of debutanized gasoline and B-B cut. The reactor change is a high-boiling tar separator overhead of the following characteristics:

Init.	430° F.	30%	675	90%	875
5%	525	50	730	95	915
10%	575	70	785	EP	1000+

Figure 4A shows a typical temperature pattern obtained during a complete cycle in a laboratory pilot unit similar in tubular arrangement to conventional commercial reactors, and where accurate temperature measurements are taken. The chart shows that the rate of combustion is rapid as indicated by the fast increase of temperature during regeneration and that the progression of combustion through the bed occurs in a fairly narrow zone as the maximum temperatures are attained at the end of the bed after combustion is complete at the front. It also shows temperatures attained at the top of the fins having the design shown in Figure 2A.

On Figure 4B are plotted the rate of heat liberation as measured by the analysis of the regeneration flue gas and the rate of heat absorption as measured by the temperature differential between catalyst and cooling medium. At their point of intersection the area between the two curves represents the total heat absorbed by the catalyst and the finned elements. This heat is released subsequently during the

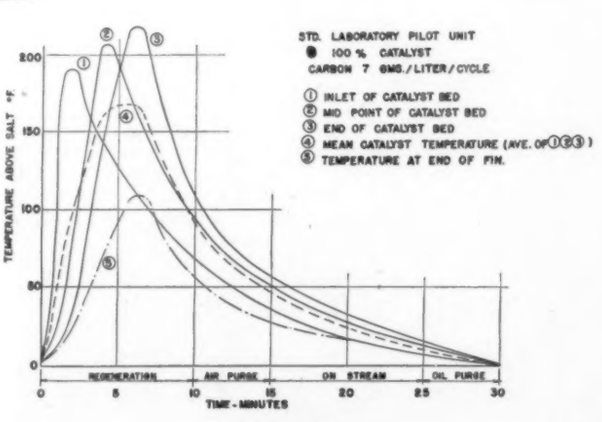

FIGURE 4-A

Typical temperature pattern, conventional design.

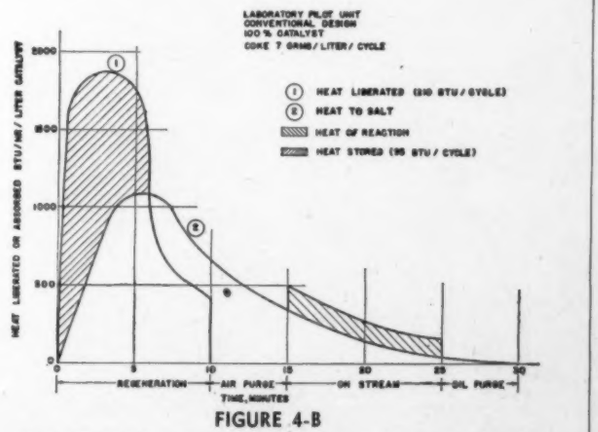

FIGURE 4-B

Heat liberated and absorbed during cycle, conventional design.

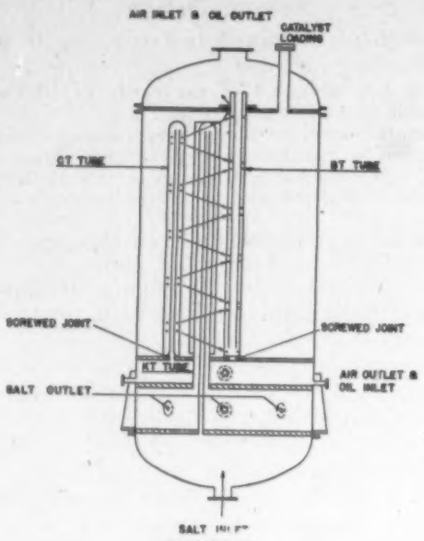

FIGURE 5
Longitudinal section, modified design.

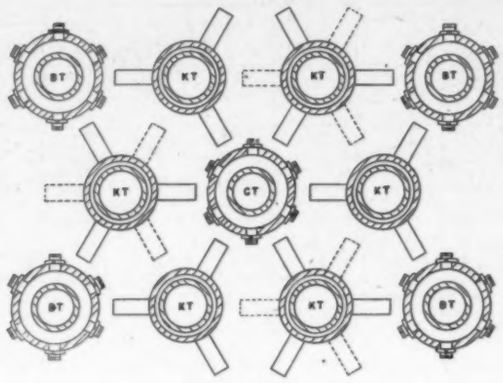

FIGURE 5-A
Typical tube pattern, modified design.

air purge and the on-stream period by the salt and also the heat of reaction.

The analysis of heat transfer in the conventional reactor and the study of the difficulties encountered in maintenance and changing of catalyst have resulted in the development of a new simplified design of the following characteristics:

1. Cooling surface substantially decreased and interlocking of fins eliminated to insure free flow of catalyst. See Figure 5. As seen by the temperatures indicated in the diagram, the peak and average temperatures of the fins are lower than formerly, thus the rate of oxidation is materially reduced.
2. Heat capacity of catalyst increased by mixing the pellets with a granular, chemically inert, cheap material of high volumetric heat capacity, which is easily recoverable during catalyst changes. The effect of this chemically inert material is to limit the temperature rise of catalyst during regeneration and the temperature fall during on-stream periods.
3. All tubular elements conveying air and oil vapors to and from the catalyst space easily removable, being assembled by a threaded joint in the lower tube sheet. In the former design the air outlet tubes were welded to their respective salt tubes (D.T.) and hence their inspection and cleaning necessitated dismantling the reactors, including the rolled and welded joint of the salt tubes.

Figure 5 shows the general arrangement of the new design as used in one of the Sun Oil Company units which has been in operation for the past year.

Figures 6A and 6B show the plot of temperatures during the cycle and the respective rate of heat release and heat absorption.

By comparison with the similar plots of Figure 4A and 4B, the following conclusions may be drawn:

1. The overall rate of heat transfer being decreased, the differential temperature between catalyst and salt is higher during the on-stream period. This factor is corrected by adjustment of the other operating variables such as salt temperature and on-stream pressures.
2. At the same carbon burn-off, the temperature differential during the regeneration period is lower, due to the increased heat capacity of the system.
3. The effect of the variation of heat transfer and heat capacity is shown by the large increase in heat absorbed in the catalyst during the regeneration period.

The experience of the first year of operation has led to certain additional improvements which are now incorporated in the latest units built, as follows:

1. Addition of fins on one half of the K.T. tubes (shown in dotted lines on Figure 5A) resulting in an increase of coke-burning capacity, all of the advantages of the design remaining.
2. Replacement of the packing insuring tightness of the top tube sheet against the tubular elements carrying oil and air to and from the top of the cases to the catalyst space by a stainless steel bushing providing enough clearance for passage of part of the air and vapors. Adjustment of orifice sizes governing the flow through the upper bed is made to insure proper distribution. With this scheme, the time of removing packing, as well as cost of replacement are eliminated.

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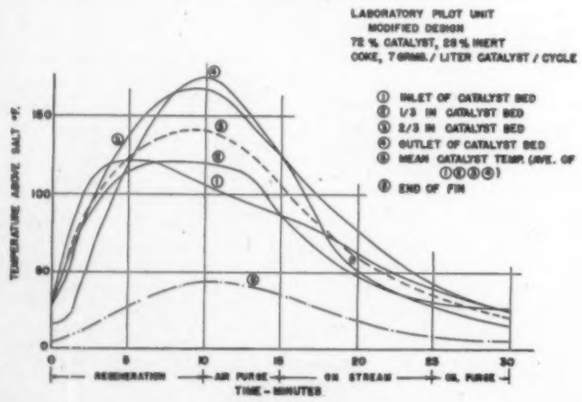

LABORATORY PILOT UNIT
MODIFIED DESIGN
72 % CATALYST, 28 % INERT
COKE, 7 GRMS. / LITER CATALYST / CYCLE
① INLET OF CATALYST BED
② 1/3 IN CATALYST BED
③ 2/3 IN CATALYST BED
④ OUTLET OF CATALYST BED
⑤ MEAN CATALYST TEMP. (AVE. OF ①②③④)
⑥ END OF FIN
TEMPERATURE ABOVE SALT °F.
REGENERATION
AIR PURGE
ON STREAM
OIL PURGE
TIME - MINUTES

FIGURE 6-A
Typical temperature pattern, modified design.

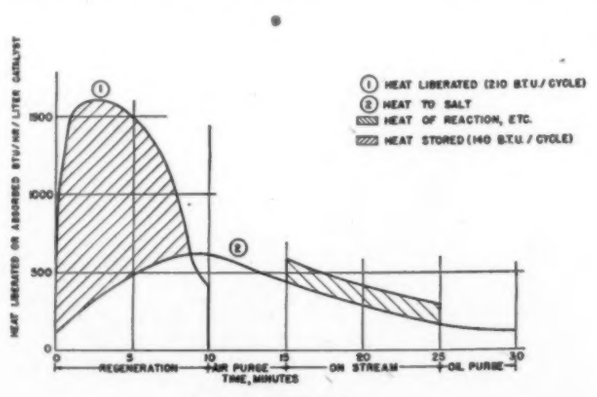


FIGURE 6-B
Heat liberated and absorbed during cycle, modified design.

DESIGN OF
Concrete Piers for Horizontal Storage Tanks

O. L. GARRETSON and HARRY R. ZEIGLER, Chemical Engineering Department
Phillips Petroleum Company, Bartlesville, Oklahoma

THE introduction and practically exclusive use of fusion-welded tanks for liquefied petroleum gas has made possible certain improvements in design of piers for horizontal pressure vessels.

The steel in pressure vessels is subjected to twice as much hoop stress as longitudinal stress. This makes possible the use of comparatively light girth seams in riveted vessels, but fusion-welded seams are of the same strength regardless of direction. Due to this fact girth seams of riveted tanks cannot have additional stresses imposed on them, and it has therefore been necessary to provide a separate support for each ring. Each pier presents a problem in prevention of rust or corrosion as well as in general neatness, such as grass and weeds trimming and cutting. It is also a fact that building piers so that they carry their full portion of the load is difficult and seldom accomplished. When the principal load is carried by the central pier or piers the tank has a tendency to go out of round.

In the case of fusion-welded tanks it is possible to use two piers, a very desirable arrangement for reasons mentioned above, and this in turn makes improved pier designs possible.

It has been shown* that horizontal tanks if held round at the supports will stay round between supports under all conditions of loading, practically no circumferential bending stresses are set up and longitudinal bending stresses can be computed by ordinary beam formulas.

Some forward-looking companies were quick to take advantage of the benefits to be gained by supporting propane and butane tanks as beams, limiting the supports to two piers and locating the piers as close to the tank heads as possible in order to prevent deformation of the tank shell.

In addition to the reduction of maintenance costs, this change resulted in reduction of first costs. A typical case, that of a 30,000-gallon propane tank, showed an apparent saving of two thirds in dirt excavated, three fifths in the amount of concrete required, and three fourths in surface area of forms which had to be built. Savings such as the above are based on conventional design of old style piers with extremely low soil bearing pressures with the usual spread footings, but even if the spread footings are eliminated on piers of conventional design the two-pier method will still show important savings.

In order to conform to safety standards set up by the National Board of Fire Underwriters the capacity of propane and butane tanks must not exceed 30,000 gallons. Practical consideration such as economy of design, maximum length or diameter which can be shipped, etc., control the proportions of these tanks. Ordinarily the length-diameter ratio of an 80-pound-working-pressure butane tank will not exceed 5 to 1 and for a 200-pound-working-pressure propane tank this seldom exceeds 9 to 1. For tanks which do not have greater length-diameter ratios than these the unsupported length between two piers located near heads will not cause excessive stresses.

An adaptation of the two-pier support method is to place piers at the quarter points of the tank, thus causing the tank to overhang at each end. This is not desirable as the tank heads are not in the proper location to maintain the roundness of the tank.

The safe distance between supports for a tank sup-

* Design of Large Pipe Lines by Herman Scherer: Transactions of the American Society of Civil Engineers, September, 1931.

ported near its extremities can be determined from the flexure formula for a simple beam,

$$M = \frac{wl^2}{8}$$

where M is the bending moment
w is the load per lineal inch
l is the unsupported length in inches

Since the desired length will be in feet this formula can be written

$$M = \frac{144\,wL^2}{8}$$

where L is the unsupported length in feet

Also $M = fS$ where f is the extreme fiber stress and S is the section modulus

$S = 3.1416\,r^2t$ (for a thin walled cylinder) where r is the inside radius and t is the thickness.

Then

$$\frac{144\,wL^2}{8} = 3.1416\,fr^2t \text{ or } L = \frac{r}{12} \times \sqrt{\frac{8 \times 3.1416\,f\,t}{w}}$$

For practical purposes this can be written

$$L = \frac{5r}{12} \times \sqrt{\frac{ft}{w}}$$

Values of r, t and w are given in Tables 1 and 2

Due to allowance for joint efficiency and thickness of metal, joints of ASME-U69 tanks can be stressed to 8752 psi in the case of 55,000 steel and 11,080 psi for 70,000 steel. Since only half of this allowable stress is imposed on the girth seams by the internal pressure, the remaining half is available for resistance to bending. Thus the value of "f" for a 55,000-pound steel tank is 4386 psi, and for a 70,000-pound steel tank it is 5540 psi. By substituting these "f" values and the values in Tables 1 and 2 in the formula

$$L = \frac{5r}{12} \times \sqrt{\frac{ft}{w}}$$

the limiting unsupported tank lengths can be found. These lengths are shown in Table 3. In calculating the unit weights in Tables 1 and 2 the tanks were assumed to be full of water instead of containing 42 percent of the water weight in the case of propane or 50 percent of the water weight in the case of butane. This represents the condition during a hydrostatic test and it is therefore obvious that the lengths in Table 3 are conservative for these tanks when operating conditions prevail.

Table 4 shows the relative diameter and length for tanks of various capacities. The lengths in this table are the tangent lengths. The lengths in the table are for tanks with elliptical heads and if flanged and dished heads are used the tangent lengths will be slightly greater.

Figure 1 shows graphically the relation between Tables 3 and 4. A glance at this figure is enough to show that propane and butane tanks of good proportions can be safely supported on two piers.

The height of tanks above ground will depend on the purpose of the installation, and the ground conditions. When the bottom outlet of a tank is used, the tank should be high enough to make the shut-off valve readily accessible. When this outlet is connected to the suction side of a pump the tank should be high enough to provide a good static head on the pump. For most other operations it is satisfactory to have the tank close to the ground.

In the case of low piers it is not necessary to use vertical reinforcing but in the case of piers 18 inches or more above ground line some vertical reinforcing should be used. This does not have to be "designed" but a small amount of "random" reinforcing will serve. The change in length of a tank 60 feet long due to 100 degrees difference in temperature will be approximately one-half inch. This change in length will ordinarily be due to a change from summer to winter or winter to summer conditions and therefore will occur very slowly. For this reason it will probably not exert very much thrust, but vertical reinforcing in taller piers will be valuable in preventing damage due to this change in length.

The lengths of piers should be equal to or more than the outside diameter of the tank which is placed upon it. The footing will be the same length as the pier and will have to be spread sufficiently to transfer the load to the soil in accordance with local conditions. The soil load should be based on the combined weight of the tank, its full content of water and the weight of the concrete. Unless the soil is known to have a very low bearing capacity it can be assumed

TABLE 1

Thickness of Steel and Unit Weight for Butane Tanks 80 psi working pressure, 55,000 psi Ultimate Strength Steel, Par. U 69, ASME Code.

Inside Diameter, Inches	Radius Inches	Thickness Inches	Weight Lbs. per Lineal Inch		
			Steel	Water	Total
48	24	.220	9	64	73
60	30	.274	14	102	116
72	36	.329	21	146	167
84	42	.384	29	200	229
96	48	.438	38	260	298
108	54	.494	48	330	378
*120	60	.548	59	408	467

* Practical shipping limitation.

TABLE 2

Thickness of Steel and Unit Weight for Propane Tanks 200 psi working pressure, 70,000 psi Ultimate Strength Steel, Par. U 69, ASME Code

Inside Diameter, Inches	Radius Inches	Thickness Inches	Weight Lbs. per Lineal Inch		
			Steel	Water	Total
48	24	.433	19	64	83
60	30	.542	29	102	131
72	36	.650	42	147	189
84	42	.758	57	200	257
96	48	.868	75	260	335
108	54	.975†	94	330	424

† Practical thickness limitation is 1 inch.

TABLE 3

Safe Unsupported Lengths of Fusion Welded Tanks Par. U 69, ASME Code

Inside Diameter, Inches	Length Between Supports, Feet	
	Butane Tanks	Propane Tanks
48	36.1	52.1
60	40.4	59.8
72	43.9	65.5
84	47.5	70.7
96	50.6	75.2
108	53.8	80.0
120	56.8	....

TABLE 4

Tangent Length in Feet for Tanks of Various Diameters and Capacities

Inside Diameter, Inches	Gallons 2 Heads	Gals./LF in Shell	Gross Capacities of Tanks, Gallons					
			30,000	25,000	20,000	15,000	10,000	5,000
			Tangent Lengths, Feet					
48	126	94	....	....	....	....	....	52‡
60	244	147	....	....	....	....	....	32
72	424	212	....	....	....	....	45	22
84	676	289	....	....	66‡	50	32	15
96	1000	376	....	64‡	51	37	24	....
108	1428	475	60	50	39	29	18	....
120	1958	590	48	39	31	21	14	....

‡ Denotes lengths of butane tanks unsuitable for support by two pier method. These lengths are not exclusive for propane tanks.

capable of supporting 3000 pounds per square foot and the spread of the footing can be calculated accordingly. The thickness of this footing should be made sufficient to withstand shear unless a very thin section is desired, in which case it must be reinforced. The conservative allowable shearing stress for concrete is 100 pounds per square inch. One pier for a

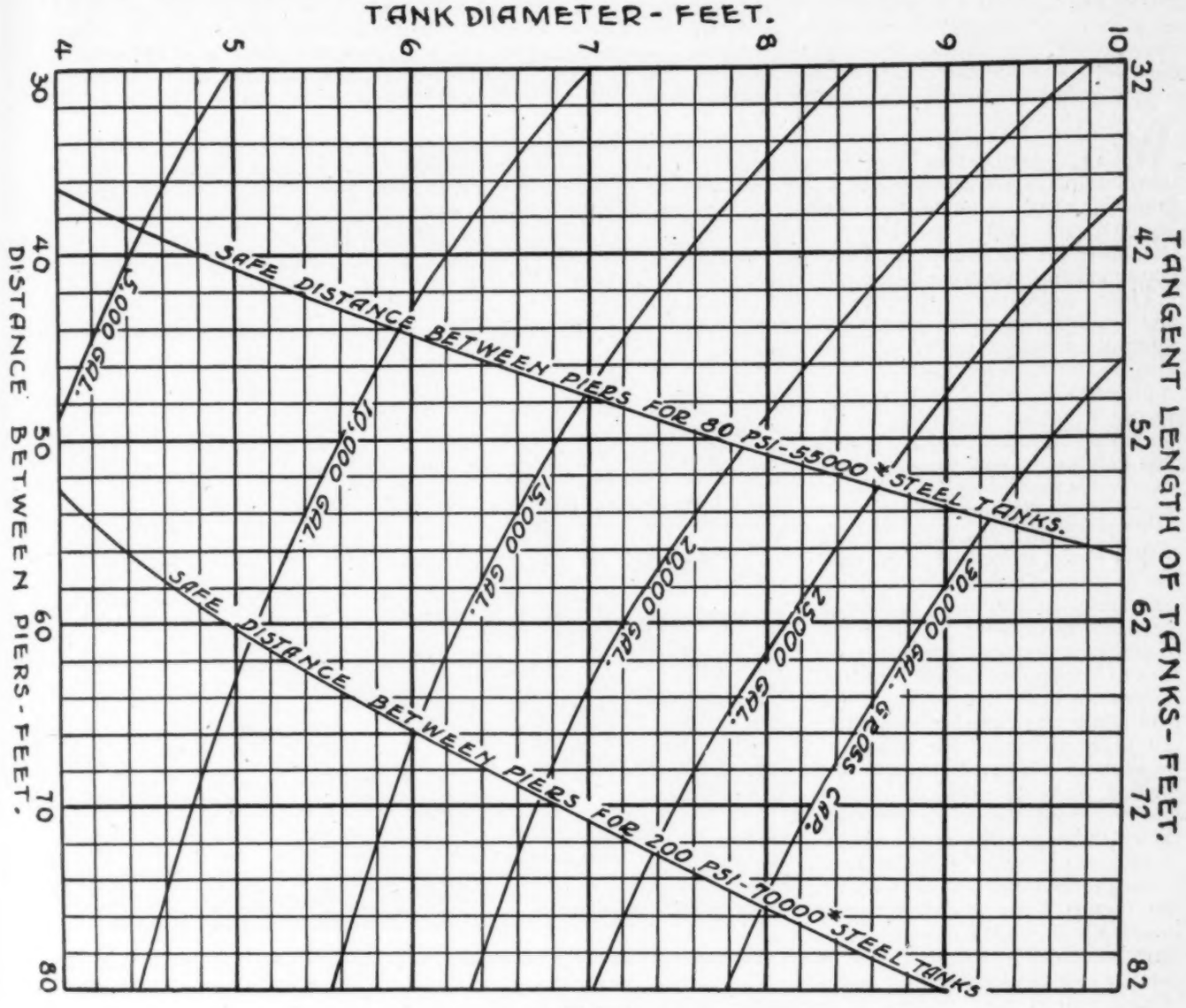


FIGURE 1

Pier spacing and tangent lengths for tanks of various capacities and diameters.

(ASME Par. U69. Construction with Elliptical Heads)

should be bonded together in the usual manner by means of steel dowels or strong pieces of stone.

Frequently piers split just under the center line of the tank. In some cases the split appears to be widest at the top of the pier and in other cases it appears to be widest at the ground line. Failure of this kind seldom causes anything more serious than an unsightly condition. However, there are means of preventing it and they should be used. One means is to provide for a resilient cushion between the tank and the pier. This can be made of three-quarter-inch expansion-joint material such as is commonly used in paving work and this serves the further purpose of retarding rusting and corrosion of the tank to a very great extent. To provide for expansion-joint material the shape of the tank saddle should be constructed to a radius three-quarters inch greater than the outside radius of the tank itself.

Further and most positive insurance against splitting of the pier will be by means of random reinforcing placed horizontally near the top and near the bottom of the vertical section of the pier.

30,000-gallon propane tank, 106 inches ID, 60½ feet long will carry 21 tons of steel and 62½ tons of water or approximately 85 tons total. The footing will be in double shear and the thickness will be

$$\frac{85 \times 2000}{100 \times 108 \times 2}$$

from which we find the required thickness will be about 8 inches. Ordinarily these footings are made 12 inches thick for small tanks as a minimum and for large tanks this is usually 15 inches thick and it is therefore evident that reinforcing against shear is unnecessary in most cases. However, a few "random" reinforcing rods are recommended.

The vertical portion of the pier and the footing

Mississippi River Clarification Plant Provides

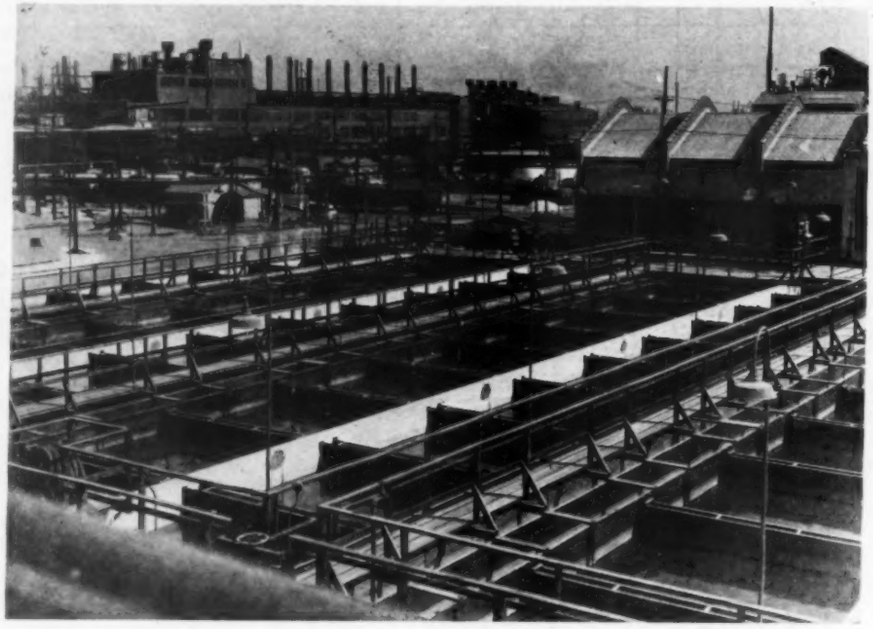
Large Volume of Clear Water at High Rate of Flow

GILBERT F. MOORE, Power Supervisor
Ethyl Corporation, Baton Rouge, Louisiana

WATER clarification by sedimentation and coagulation involves processes familiar to many engineers; however, usual sedimentation basins are remembered as extremely large structures requiring long retention time for the water to clarify and with effluent water usually requiring further filtration.

The consolidation of the coagulation, sedimentation and filtering procedures in one operation by means of specially designed units to obtain clear water in large quantities at high rates of flow is a relatively recent development. Such an installation designed to handle 20,000 gallons per minute was made at the Baton Rouge, Louisiana, plant of Ethyl Corporation.

This plant, manufacturing Ethyl Fluid, is located in the center of a rapidly growing chemical and petroleum-refining industrial section. As in most chemical and refining industries, large quantities of cooling water are required in process heat exchangers. When the plant was first built in 1937, all make-up water for this purpose was obtained from wells drilled and screened at depths varying from 225 feet to 2000 feet. With the expansion of the industrial area, increasing withdrawal from the underground water supply resulted in lower pumping levels until in 1944 it was necessary to develop a new source of supply independent of the ground water, and it was decided to use water from the Mississippi River, which flows adjacent to the plant, to supplement the wells.



Four precipitator basins in the Baton Rouge water clarification plant each handle 5000 g.p.m.

As a result of studies instituted at that time, a modern water-clarification plant was installed to remove the mud from the river water brought into the plant. An idea of the amount of mud removed may be realized when it is considered that the turbidity of the raw river water, expressed as parts per million of silica which would give equal capacity to distilled water, averages between 300 and 1000 parts per million, while the water after clarification has an average turbidity of 2 parts per million. This represents a daily removal at maximum capacity of 700 tons of mud. This turbidity removal is accomplished very rapidly. The total water storage capacity of the precipitator basins is only approximately 1,500,000 gallons, which, at maximum flow rates of 20,000 gallons per minute, allows a retention time of only 80 minutes.

Clarification of water by sedimentation alone has definite limits which are set by the particle sizes to be removed and is limited roughly to the elimination of sands and silt. Particles in the size range immediately below that of sands and silt require impractically long settling times while those in the colloid range cannot be removed at all by sedimentation due to the electrostatic charge on their surface, which, being the same on all particles, prevent settling by the repelling effect of one particle on the other.

Coagulation, the process of agglomerating the fine particles, is accomplished by adding a chemical which in solution furnishes electrical charges of opposite polarity to neutralize the charges on the colloids and permit them to come together. Also the coagulants form a floc or gel precipitate, enmeshing the finer particles into large groups which can settle out in a practical length of time because of thier greater mass.

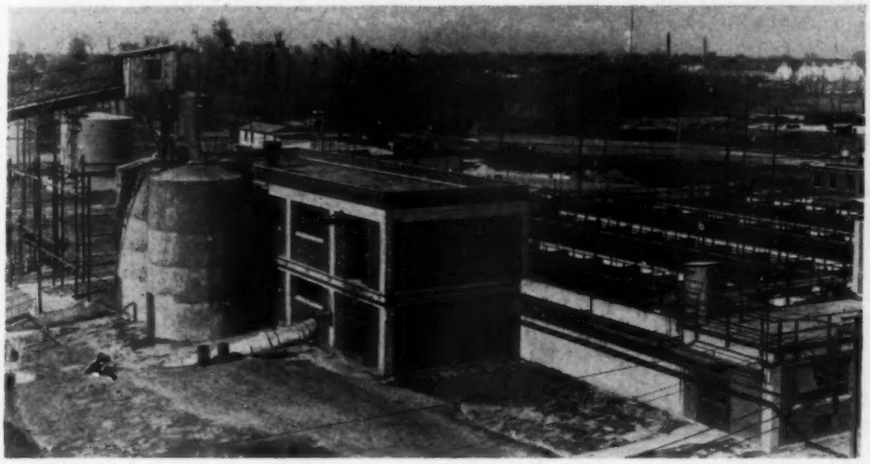
In the permutite precipitator the above processes are combined but the floc is not permitted to settle. Instead, by mechanical agitation and hydraulic flow it is kept in suspension until it forms a sludge blanket through which all further water must be filtered, thus resulting in the removal of not only practically all of the turbidity but also the small amount of very fine floc. The effluent water is crystal clear.

The process, as installed, may be followed by the

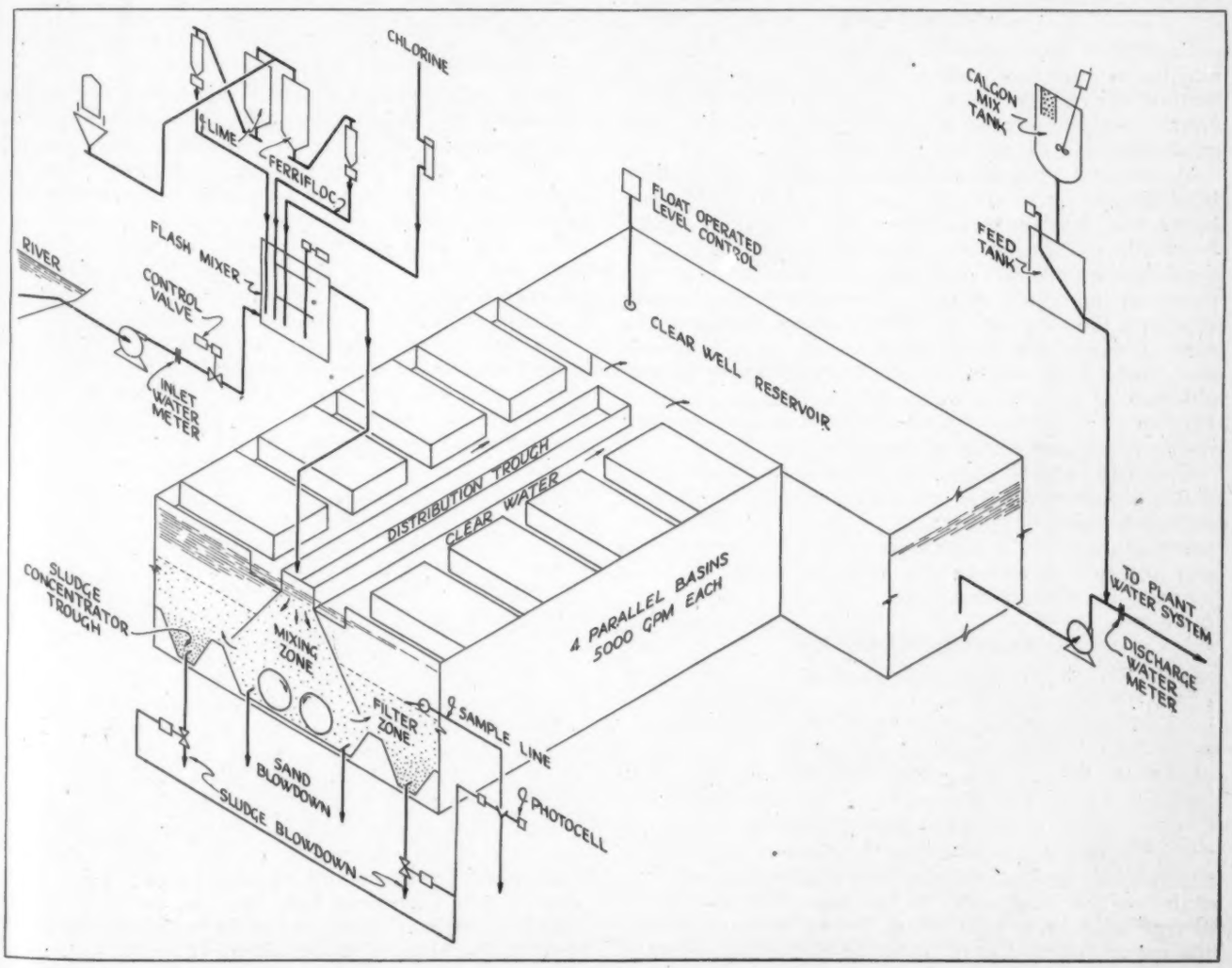
included flow sheet. The river water and chemical solutions are mixed in a rapidly agitated flash mixing chamber, then, led by flumes and sluice gates to four parallel horizontal-type precipitator basins, each having a capacity of 5000 gallons per minute. The treated water in which the floc is beginning to form flows downward into the mixing zone where the slow-rolling agitation by the paddle agitator allows the new floc to agglomerate or grow with the floc already present. From the mixing chamber, the water passes out into the filter zone where, due to the gradually increasing cross-sectional area, the velocity of the water gradually decreases. As the water slows down, the floc particles finally become too heavy for the decreased velocity of the water to support them, and they remain suspended to form a sludge blanket through which following particles are filtered. The top level of this blanket is dependent upon the rate of flow of the water and is sharply defined with clear water above it, and heavy sludge below it. As the sludge particles become larger and heavier, they settle down against the flow of water into the mixing zone to furnish nuclei for the new floc to form with. As a consequence, there is a continuous flow of suspended particles out of the mixing zone into the filter zone and counterflow back. This results in maintaining approximately the same concentration of sludge throughout the filtering and mixing zones and obtains maximum contact of the active sludge with the water, resulting in reduced quantities of coagulant chemicals required.

In order to draw off the excess sludge with as little waste of water as possible, a sludge concentrator is formed at the bottom of the vee, where collecting blow-off pipes are installed.

Automatic operation of the



This photograph is a general view of Ethyl Corporation's plant for clarification of Mississippi River water at Baton Rouge, Louisiana, with chemical handling equipment in the foreground. Below is a flow diagram of the plant.



sludge removal is obtained by locating a swing sample pipe in the area of the top of the sludge blanket so that a water sample passes continuously before a photocell controls the automatic blow-off valve. As the sludge level rises in the basin, the sample water darkens and the photocell relay opens the sludge line blow-off valve for a predetermined interval. To prevent too rapid operation of the blow-off valve, a time-delay switch is interposed in the circuit to allow a short time for settling of the blanket before a repeated blow-off.

Because of the large area of the top of the sludge blanket, varying flow rates cause very little changes in the level of the sludge blanket and fairly rapid changes in flow can be absorbed without breaking up or losing the floating filter blanket. Individual precipitator basins have been run at flows ranging from 1000 to 6000 gallons per minute with approximately constant results.

The clear water leaving the sludge blanket is collected in lateral troughs from which it passes to the two central flumes and is discharged into a clear-well reservoir common to all four parallel basins. From this reservoir, the water is pumped into the plant water-distribution system.

The coagulant used in the water clarification is a water-soluble ferric sulfate. The average dosage varies between 30 and 40 parts per million.

The pH of the dosed water is maintained between 8.5 and 9.0 by the addition of lime to obtain the best range for coagulation. The average dosage of lime varies between 20 and 40 parts per million.

Chemical Dosage

Control of the coagulant dosage is maintained primarily by close control of the pH of the treated water leaving the flash mixing chamber which is continuously indicated and recorded by means of a calomel electrode cell pH recorder.

Both the coagulant and lime are fed by dry feeders into dissolving tanks and put into solution before being added to the raw water. The coagulant feeder is of the oscillating-tray type with the amount of feed determined by changing the amplitude of the stroke of the feed tray. The lime feeder is a vibrating type and the feed rate is determined by changing the rate of movement of a counterpoise on a balanced scale supporting the feed bin. All raw water is prechlorinated to a chlorine residual of approximately 1.0 part per million in order to prevent algae growth in the precipitator basins and plant pipe lines.

Corrosion and scale formation throughout the plant water piping system is controlled by the addition of sodium hexameta phosphate, to the extent of 2 parts per million. This inhibitor is added by means of a wet solution feeder to the clarified water as it is pumped from the plant.

Automatic Operation

Nearly full automatic operation of the plant is obtained by the use of the above equipment for controlling the sludge removal and by having the feed rates, once established, controlled by a flow controller in the raw-water-inlet line. These automatic controls function as follows:

The flow of water as required from the plant, together with a level control in the clear-well reservoir, control the position of the inlet raw-water valve to maintain the inlet water at the same rate as the discharge and keep full levels in the reservoir, thus making up for the loss of water through sludge removal.

The inlet-water flow integrator energizes the motor drive on the coagulant oscillating feed tray and the motor drive on the movable counterpoise on the lime feeder in proportion to the rate of flow, so that once the feed rate in parts per million is set on the feeders it is maintained independent of the quantity of water required.

In a similar manner, the sodium phosphate feeder motor is actuated by the integrators on the discharge water line flow meters to control the addition of the inhibitor to the clarified water.

Few Adjustments Required

By the use of these automatic controls, operating labor required is kept at a minimum. The plant operates on a 24-hour, 7-day basis with one operator required on the day shifts to fill all feed bins and make routine adjustments, and a part-time operator on the night shifts who checks the plant every four hours to insure good operation. Feed rates are determined and set by means of laboratory floc tests and very few adjustments are required.

Chemical analyses are run on inlet and effluent water once a day and determinations are limited to the following which are representative of average conditions:

	River Water	Clarified Water
pH	7.8	8.3
Turbidity, ppm	500	2
Phenolphthalein Alkalinity	0	4
Methyl Orange Alkalinity	65	68
Hardness (Soap)	84	102
Calcium Hardness	66	76
Chlorides	16	20
Free CO_2	2	0
SO_4	24	40
Suspended Solids	434	10

Ferric sulphate and lime are purchased in box-car lots which are unloaded into a track hopper by means of a power shovel. They are then conveyed through an elevator and inclined conveyor to two concrete storage silos having conical bottoms discharging into smaller individual elevator-conveyors to the individual feed bins located in the chemical building. Both the outside storage silos and the inside feed bins are equipped with level indicators which sound an alarm and cut off the conveyors when full.

The sodium phosphate is purchased in bag lots and is dissolved in an agitated mixing tank before dropping to the solution feeders. Since sodium hexameta phosphate is corrosive in strong solution, approximately 10 percent by weight of soda ash is added, when dissolving, to limit corrosion on the solution tank and equipment.

Chlorine is manufactured on the plant and piped to the clarification plant where it is fed into the raw water in the flash mixing chamber by means of a chlorinator having a maximum capacity of 2500 pounds per day.

Costs

Average raw material costs expressed in dollars per 1000 gallons of water treated are as follows:

Ferric sulphate	$.006
Lime	.001
Chlorine	.001
Sodium hexameta phosphate	.001

This plant as installed was designed by The Permutit Company and, while the basic design remains unchanged, its shape and size can be varied considerably to suit individual installations. In many cases, existing settling basins have been adapted to this type of operation with satisfactory results.

Pilot-Plant and Commercial Desalting

M. J. SEE, J. P. LINDAHL, H. R. TALIAFERRO and
C. R. HARTE, JR.
Standard Oil Company (Indiana)

IN LATE 1944 Whiting refinery started running a substantial amount (45,000 barrels per day) of West Texas crudes containing up to 300 pounds of salt per 1000 barrels Previously, the refinery had operated almost exclusively on Mid-Continent crudes containing about 40 pounds per 1000 barrels, and the refinery equipment had been designed for this quantity of salt. With the advent of high-salt crude, desalting measures became necessary. Equipment was provided to desalt the 45,000 barrels per day of West Texas crude; and a pilot plant was set up for study of desalting processes.

The commercial desalting equipment now operating at Whiting includes four units: a desalter for a 13,000-barrel pipe still, and desalters for three continuous shell still crude batteries with a total capacity of 27,000 barrels per day. All the desalters are of the coalescer and settler type, and are quite similar in design. Figure 1 presents a flow diagram of a typical desalting unit, at a 10,000-barrel-per-day crude battery.

As is shown in Figure 1, the process consists essentially of mixing a small amount of water with the crude, heating the mixture to about 230° F., coalescing the microscopic water droplets on a collecting surface such as excelsior, and settling at a pressure sufficient to prevent vaporization. Cold crude is mixed with demulsifying agent at the suction side of a crude-charge pump. Water from either condenser boxes or the service water line is added on the discharge side of the crude pump. The mixture of oil and water passes through mix valves located just

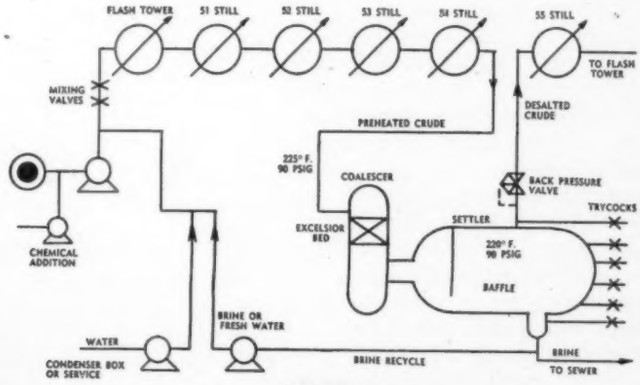
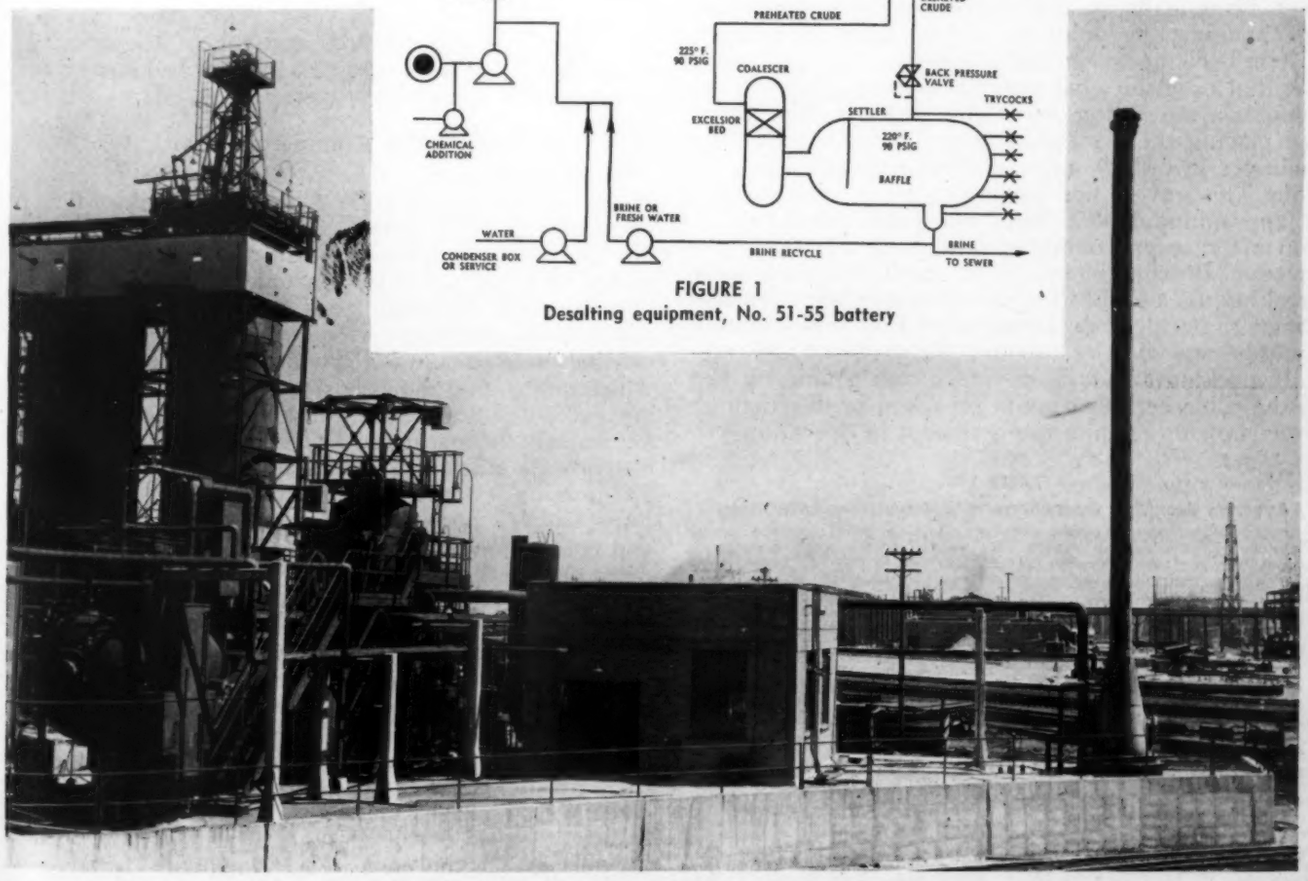


FIGURE 1
Desalting equipment, No. 51-55 battery



Commercial desalting unit at No. 6 pipe still, Whiting refinery. Shown, left to right, are the settling drum (horizontal), coalescer (vertical), pump house, and blowdown stack. The two vertcal cylinders in the background are heat exchangers connected with the pipe still.

downstream of the crude-charge pump and then through the tower coil exchangers to the coalescer. The pressure drop across the mix valves is held at a maximum of 30 psi as limited by crude-charge-pump head. Little or no pressure drop occurs in the exchanger system. The oil-water mixture enters the coalescer at 225° F. and a pressure of 90 psig.

The major vessels of the desalting unit are the coalescer, a 15-foot vertical drum 7.5 feet in diameter, and the settler, a 24-foot horizontal drum 8 feet in diameter. Under present operating practice the 5.5-foot bed of coalescing medium is gum-free excelsior. The coalescer is connected to the settler by means of a 36-inch pipe, the large size being used to prevent reemulsification of the oil and water. In entering the settler the coalesced material passes underneath a baffle which causes the oil to be washed by the brine as it enters the settling zone. The brine level is therefore maintained above the lower edge of the baffle.

Final separation of crude and brine takes place during a 30-minute holding time in the settler. The desalted crude leaves the top of the vessel under control of a back-pressure valve which maintains sufficient pressure in the system to prevent vaporization. From this point the crude resumes its normal flow through the crude distillation unit. The brine is drawn off the bottom of the settler at a rate regulated by a level-control instrument.

During the 1½ years since initial operation of these units, only minor changes have been made in the process. Some improvement has been made in the technique of packing excelsior into the coalescers, and certain changes have been made regarding the position of the mixing valves. Also, minor changes have been made in the temperature of coalescing, by changing the point at which the crude-preheat system is connected to the coalescer.

Initial excelsior coalescer packs were prepared by removing the baling wire from the excelsior bales and placing the pressed sections of the bales in the coalescer in orderly piles, packed as tightly as possible. This gave a bed packed to an average density of approximately 8 pounds per cubic foot. Inspection after several months' operation indicated that severe channeling had taken place, that the beds were breaking up, and that the excelsior was being washed out of the coalescers. It appeared that this situation could be remedied by unbaling the excelsior and carefully packing and tamping it to form a uniform bed in the coalescer. Two units repacked in this manner have shown a definite improvement in operation.

TABLE 1

Average Desalting Operations with Demulsifier at Whiting

Charging West Texas crude. Water addition point: Crude pump discharge. Pressure drop across mix valves and exchangers: 10 to 120 psi. Temperature of crude to desalter: 225 to 275° F. Pressure on desalter system: 90 to 135 psi.

	Demulsifier Added Gals. Per 1000 Bbls.	Vol. Percent Water Added	CRUDE IN		CRUDE OUT		Brine Lbs. Salt Per Bbl.	Salt Removal, Percent
			Salt, ptb	B.S.&W., Percent	Salt ptb	B.S.&W., Percent		
June, 1945	1.2	5.1	251	0.6	38.5	0.9	3.8	84.5
July	1.2	5.1	226	0.5	26.2	0.7	3.8	84.8
August	1.2	5.0	196	0.4	33.8	0.8	3.4	82.9
September	1.4	4.9	192	0.6	37.1	1.0	3.3	80.6
October	1.4	4.9	179	0.5	28.8	1.3	3.4	83.9
November	1.4	5.1	200	0.5	30.2	1.3	3.5	84.8
December	1.4	5.4	252	0.6	40.5	1.3	4.3	84.0
January, 1946	1.5	5.4	261	0.7	36.4	1.2	4.1	86.0
February	1.4	5.7	275	0.6	32.3	1.2	4.5	88.3
March	1.7	8.5	270	0.6	31.6	1.0	3.9	88.4
April	1.2	8.6	254	0.6	23.6	1.2	3.6	90.6
May	1.2	8.6	250	0.6	25.9	1.5	3.7	89.7

The point in the system at which the oil and water is mixed has been found to have an important influence on mixing efficiency. Gentle mixing through the exchanger system is superior to violent mixing in mix valves; but exchanger pressure drop is insufficient to give enough mixing at certain units, and additional valve mixing is therefore required. When mix valves just upstream of the coalescers were found to give violent mixing too close to the coalescers, these valves were removed and on two units were reinstalled upstream of the exchanger systems; the other two units are now operating without mix valves.

Results on Commercial Equipment

Average monthly operations of the Whiting desalting units are shown in Table 1 for the period June, 1945, through May, 1946. Fairly uniform operation was maintained during this period, the gradual improvement being due to increased experience. Demulsifier addition varied from 1.2 to 1.7 gallons per 1000 barrels of crude, water addition from 4.9 to 8.5 volume-percent on crude. Salt in the incoming crude exhibited the usual seasonal variation and ranged from 180 to 275 ptb; salt content of the treated crude varied from 26 to 40 ptb. The best operation for a single unit for a 24-hour period has given a treated crude with a salt content of 17 ptb. The higher salt figures during December and January were the result of trouble with the demulsifier injection facilities during the severe cold weather. It should be noted that the salt content of the treated crude was reduced in February and March to 32 ptb in spite of the fact that this is the period during which salt content of the raw crude has reached its seasonal peak. With the proper use of the present facilities it is believed that the salt content of treated West Texas crude may be held below 30 ptb. BS&W content of the treated crude has varied from 0.9 to 1.3 percent and has given no trouble in flash tower operations.

Corrosion in Commercial Units

Since brines obtained when desalting West Texas crudes with approximately 5 percent of Lake Michigan water are acid to the extent of 3 to 4 pH, a serious corrosion problem was anticipated. It was expected that pH adjustment would be necessary and that injection of caustic or soda ash solution at the crude-pump discharge would be required. Because of the possibility of throwing down precipitates which might plug the excelsior beds in the coalescers, however, desalting operations were started without neutralization; and fortunately the corrosion problem has proved much less acute than was feared. After six months' operation, inspection of the desalting units showed negligible corrosion in the coalescers and none in the settlers except for pitting 0.01 inches deep on the bottom sixth of the vessels. Pits 0.1 inches deep were found in a strip 6 inches wide in the very bottom of the settler. Rather severe corrosion occurs in the brine drawoff lines downstream of the settler, and here lines and valves must be replaced frequently. There is no evidence of serious corrosion in the exchangers upstream of the coalescers.

Apparently the oil forms the continuous phase in the emulsion, with the droplets of brine forming the discontinous phase; and in this condition the brine does not attack the metal. Only after separation does the brine become corrosive. Adequate protection against corrosion can be obtained by installing extra metal over the bottom portion of the settlers and by

using alloy in the brine lines. Another approach now being considered is an increase in water addition. It has been found that 15 percent of water gives a brine with a pH of 6.0 to 6.5, a non-corrosive range.

Desalting Pilot Plant

In addition to the commercial desalting units there is available at Whiting a 24-barrel-per-day pilot plant for investigating various types of coalescing media and other operating variables. Pilot-plant experiments have been carried out with the objective of improving the excelsior coalescing process now employed at Whiting; and a study has also been made of a modified process in which glass fiber is used as the coalescing medium.

Figure 2 presents a flow diagram of the pilot plant, a small-scale replica of the commercial units. Crude is pumped in by means of a gear pump. Manifolding permits injection of water either before or after this pump. Mixing of crude and water is accomplished in the pump if the water is added upstream of the pump, or by applying a pressure drop across the mix valve if the water is added downstream of the pump. The

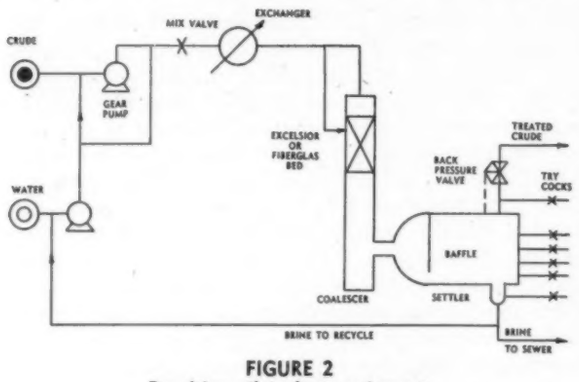


FIGURE 2
Desalting pilot plant equipment.

mixed crude oil and water are heated by interchange with steam, and pass to the coalescer, and then to the settler. Treated crude leaves the top of the settler through a back-pressure valve which holds sufficient pressure in the system to prevent vaporization.

Pilot Plant Runs Using Excelsior

A considerable number of pilot-plant experiments were made using as packing the same grade of gum-free poplar excelsior used in the coalescers of the commercial units. The excelsior was packed to a depth of 6 feet and a density of 7 pounds per cubic foot. The depth of the beds in the commercial units is 5½ feet, and the density is 5 to 7 pounds per cubic foot.

Since it was recognized that the amount of water added and the intensity of mixing this water with the crude exert a considerable influence on desalting efficiency, these variables were selected for pilot-plant investigation. Also, since the cost of demulsifying agent is a major item in the total cost of desalting, the pilot-plant experimentation was done without use of a demulsifying chemical. The results of this program are shown in Figure 3, which reveals the following facts:

(1) For a given mixing intensity, an increase in water addition results in improved desalting up to a certain water addition rate or "break-point."

* See C. G. Kirkbride, "Desalting of Petroleum With Fiberglas Packing," Petroleum Refiner, 25, July, 1946, p. 313.

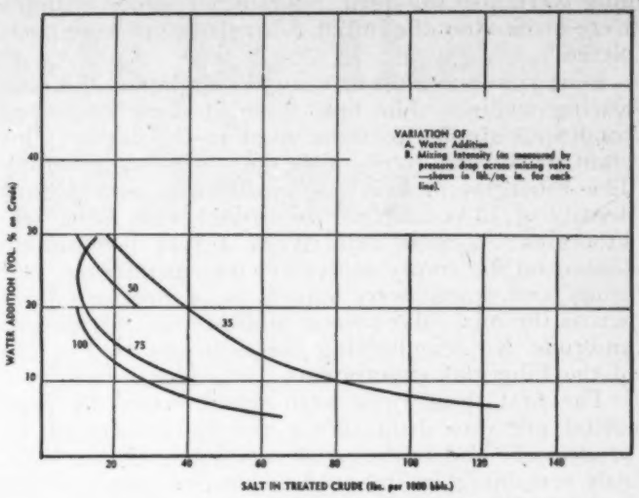


FIGURE 3
Salt content of treated crude, desalting pilot plant, excelsior packed coalescer.

(2) This break-point has been determined for the 75 psi mixing intensity only; but the shape of the curves at the other mixing intensities suggests that more water would result in poorer desalting.

(3) The greater the mixing intensity, the less water needed to obtain a given degree of desalting.

(4) Regardless of the mixing intensity and water rate employed, the best desalting leaves 10 ptb of salt in the treated crude.

On the basis of this pilot plant work it may be concluded that good desalting with excelsior as the coalescing medium can be accomplished in the absence of a demulsifying chemical. It would appear that optimum water addition and mixing intensity could be applied on the commercial units to give desalted crudes which would compare favorably with the results when using a demulsifier (20-40 pounds of salt remaining in the treated crude).

Glass Fiber as the Coalescing Medium

Attention also has been directed recently to the use of glass fiber as a coalescing medium for desalting crude oils. Ownes-Corning Fiberglas Corporation is interested in this application of glass, and has sponsored a fellowship at Texas Agricultural and Mechanical College* to encourage study of glass fiber for desalting. The results have been so encouraging that Standard Oil Company (Indiana) worked out a cooperative program with Owens-Corning Fiberglas Corporation to explore further the potentialities of Fiberglas in the Whiting desalting pilot plant.

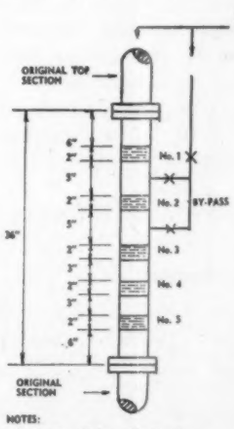

HOT FEED TO COALESCER
ORIGINAL TOP SECTION
No. 1
No. 2
BY-PASS
No. 3
No. 4
No. 5
36"
ORIGINAL SECTION
NOTES:
1. Extra Heavy 4" pipe used for all sections.
2. Fiberglas is supported on ⅛" steel discs drilled with ½" holes so as to have more than 50% free space. The discs are supported on lugs welded on the inside of the pipe.
3. The normal oil flow enters the coalescer at the top of the original section.

FIGURE 4
Pilot plant coalescer for Fiberglas experiments.

Only minor changes were required for Fiberglas experimentation. A section of the original coalescer was removed and a new section installed as is shown in Figure 4, and provision was made for installing beds of Fiberglas 2 inches thick. Means of by-passing the two top beds in case of plug-

ging were also provided. Some other minor changes were made after the initial Fiberglas runs were completed.

Four runs were made using Fiberglas as the coalescing medium. The first three of these employed conditions similar to those used in the earlier pilot plant work with excelsior as the coalescing medium. The Fiberglas in five 2-inch beds was packed to a density of 13 pounds per cubic foot with basic "B" Fiberglas. A crude velocity of 1 foot per minute (based on the empty coalescer) was maintained, and crude and water were mixed by a pressure drop across the mix valve; water addition was 25 percent on crude. No demulsifying chemical was used in any of the Fiberglas experiments.

The first three runs were characterized by high initial pressure drop across the beds followed by progressive and fairly rapid channeling of the beds. Salt remaining in the crude averaged about 15 ptb and BS&W was high (1.0 to 4.0 percent). On Run No. 4 the pilot plant equipment was changed to permit the water to be introduced at the suction of the crude pump, and the assembled equipment was pressure tested with kerosine. (Water used for pressure testing the earlier runs was believed to have impaired the condition of the beds.) The apparatus was heated by circulating hot kerosine before the mixture of water and crude oil was introduced, and the crude rate through the coalescer was cut to 0.5 foot per minute. Since the brine from the crude is acidic, acid-resistant "C" Fiberglas was used in this run. Water rate was continued at 25 volume-percent on crude. The operating conditions and desalting obtained in this run are shown in Table 2. It will be noted that the treated crude averaged 10.5 pounds of salt and 1.4 percent BS&W. This portion of Run No. 4 compares very favorably with the best desalting obtained in the pilot plant using excelsior and with the plant scale operations using a demulsifier.

Since it was apparent that the Fiberglas beds were essentially unchanged after 30 days of operation, it was decided to investigate the effect of higher crude velocity and lower water addition rather than continue the life test at the same conditions. Accordingly, the crude velocity was raised to 1 foot per minute and the water rate lowered to 12.5 volume-percent on crude—conditions readily obtainable on the Whiting commercial units. Results for 27 days' operation under these conditions are also shown on Table 2. The average salt content and the BS&W of the treated crude were virtually the same as at the lower velocity. After 57 days, desalting was still satisfactory. The run was terminated voluntarily, since it had been demonstrated that the service life of Fiberglas was sufficiently long to warrant a full-scale-plant trial of this new coalescing medium.

Based on the pilot-plant experience with Fiberglas at the coalescing medium it may be concluded that:

(1) The conditions of Run 4 will give desalting with Fiberglas equal to or better than the best results obtained at Whiting with excelsior and a demulsifier in the commercial desalting units.

(2) Because of its uniformity as to diameter and its availability in mat form, Fiberglas will probably prove fairly easy to install in the plant-scale equipment, and should give more uniform desalting results than can be obtained with excelsior.

TABLE 2
Pilot Plant Desalting with Fiberglas
Run No. 4

PORTION OF RUN	0 to 30 Days	30 Days to 57 Days
Crude Rate, G.P.H.	18	36
Crude Velocity Through Coalescer, Ft./Min.	0.5	1.0
Water, Vol. Percent on Crude	25.0	12.5
Crude In:		
Salt ptb	250	240
B. S. & W. Percent	0.7	0.8
Crude Out:		
Salt, ptb	10.5	11.3
B. S. & W. Percent	1.4	1.2

Crude and water mixed by passage through crude pump.
Five 2-inch Beds of acid-resistant "C" Fiberglas packed to 13 pounds per cubic foot density.

Application to Commercial Units

Pilot plant studies using excelsior as the coalescing medium have indicated that commercial desalting comparable to that now being obtained with a demulsifier could be realized by adding 20 to 30 percent of water on crude, without a demulsifier. A test to check the pilot-plant results in full-scale equipment is now in progress at Whiting. At this writing the water rate has been increased to 20 percent with noticeable improvement in desalting. Demulsifier addition has been decreased to 0.4 gallons per 1000 barrels of crude without impairment of desalting efficiency.

Summary

When Standard Oil Company's (Indiana) Whiting refinery started running substantial amounts of West Texas crude containing up to 300 pounds of salt per 1000 barrels of crude, desalting measures were necessary. Four desalting units of the coalescer and settler type are now in operation at Whiting. The process consists essentially of mixing a small amount of water with the crude, heating the mixture to about 230° F., coalescing by means of an excelsior bed, and settling at a pressure sufficient to prevent vaporization. A demulsifying chemical is employed. Operation of these commercial units has produced treated crudes of salt content averaging about 30 pounds per 1000 barrels, which corresponds to removing about 90 percent of the salt.

A pilot plant is used for investigation of various types of coalescing media and study of other operating variables. Pilot-plant work on desalting with excelsior as the coalescing medium has indicated that with increased water addition, it may be possible to obtain satisfactory desalting without a demulsifier. Since the cost of this chemical is an important item in the total cost of commercial operation, attractive savings may be possible. A full-scale trial of this process modification is in progress.

Another pilot-plant program has investigated the use of glass fiber as the coalescing medium for desalting. Pilot-plant runs have given desalted crudes with only about 10 pounds of salt per 1000 barrels. Since a satisfactory life was demonstrated, preparations are now being made for a full-scale-plant test.

Work to date has resulted in the development of two potentially attractive modifications of the Whiting desalting process:

(a) Modification of the present excelsior process, or
(b) Substitution of Fiberglas for excelsior as the coalescing medium.

Pilot-plant work indicates that these two possible modifications are competitive, and a full-scale testing program is under way to determine which is the more desirable.

This paper was presented at the technical service session of the Fourth Joint Technical Meeting of Standard Oil Company (Indiana) and affiliated companies, held May 13-18, 1946, at Edgewater Park, Mississippi.

Lubricant Tests By The Four-Ball Oil Tester

DR. C. F. KRIENKE
(German Aviation Institute)

Translated by E. J. Barth, Petroleum Technologist

SEVERAL tests on three different oils were made on a four-ball test machine—straight-run mineral oil, a 100 percent fatty oil, and a mineral oil containing elementary sulphur in solution. The results appeared to be somewhat scattered. When enough tests are made, however, wear values and seizure constants are obtained which show definite trends and are concordant.

Wear tests show that sulphur exerts an extremely favorable anti-wear action on metallic surfaces, far surpassing a mineral oil, and even fatty oils, which have been thought of as being good oils. Thus the study of lubrication includes the study of anti-wear compounds and how these are related to extreme pressure.

THE MANY TYPES of oil-testing apparati which are not standardized for determining friction, oiliness, extreme pressure properties, etc., are useful only if they are capable of giving reproducible and reliable results. The reason for so many types of oil testers appears to be that each one stresses some particular or important phase of lubrication or evaluates some special function of an oil—a situation created by the many types of uses to which lubricants are applicable, and also by a neglect to study the basic or "inner mechanism" of how oil films affect rubbing surfaces.

Various tests on lubricants, such as coefficient of friction, oiliness, etc., have up to now been poorly correlated, if at all, and each one has been the basis of some manner of testing machine which stresses some one apparently important property of the lubricant.

The four-ball testing machine developed by Boerlage[1] at Delft for Batavian Petroleum Corporation for use in testing extreme-pressure lubricants seems to be an answer to the question of whether a single test apparatus will evaluate an oil as to its prime function—to lubricate efficiently—and to tie in the results with those tests of next greatest significance. The cost of the apparatus is relatively low. By placing a rotating steel ball upon three solidly centered stationary balls below, there is obtained a set of conditions for friction testing which is quite reproducible in every test.

These considerations had therefore led us to build a similar apparatus for testing ordinary, aviation, and extreme-pressure lubricants. The apparatus had originally been evolved especially for proving the worth of one extreme pressure agent against another, and here it has shown to be very reliable. Our program called also for using this setup on ordinary motor lubricants. How much data on the latter has been accumulated by various investigators in the field of lubrication we do not know, except as we find it in the literature.

Testing Procedure

A. *Apparatus and Test-Oils.*

The apparatus built by us consists of four steel balls, three of which are the balls for points of contact—in fact, all four are actually the test pieces. Figure 1 shows the pyramid arrangement of the balls; three of the balls (lower) are held centered into a test cup by means of a conical shaped clamp set by a metallic gasket. The test-cup also acts as a reservoir for the oil to be tested. The fourth ball is held firm in a chuck, which is connected to the shaft of an electric motor by way of a flexible coupling; the motor is rated at 1400 revolutions per minute.

The whole apparatus is housed in one part with the attached motor; the lower portion is connected with a ball-and-socket pin which allows the attached lever to become movable and this can be loaded with known weights. The latter transfers the load to an upper plate which in turn carries the clamp and oil receptacle containing the three balls which are firmly clamped. The lever arrangement allows the use of a constant load with various lengths of the lever arm as well as for variable or increasing loads and which can be steadily maintained. The attached load regulator is water-cooled.

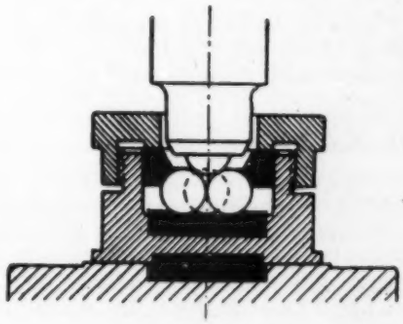


FIGURE 1
Friction elements of the four-ball tester.

During tests, the upper steel ball revolves freely, touching the three balls held securely below; this gives rise to rotational friction effects, which tend to turn the three lower balls in contact with the upper revolving ball.

The three balls and their clamp transmit their directional pull to the lever arrangement; the latter is connected to a pointer upon which the degree of pull

or frictional effect is recorded visually. The length of the pointer can be varied during a test and so set as to correspond with desired time intervals and accurate recordings are thus made available.

When the rotational deflection has reached certain values a flexible clutch arrangement on the motor is next automatically thrown out of gear and disengages the motor. The motor is then stopped and the test is completed.

It is of utmost importance that the top rotating ball be equally balanced and very accurately centered equidistantly, upon the three lower balls. Our first tests showed also the need for the spindle arrangement holding the three lower balls to be absolutely free of hindrances. Special spindles were constructed, as well as the special chuck later designed to allow the top ball plenty of play and freedom of rotation.

The test pieces or balls are precision ball bearings of ball-bearing hardened chrome steel; they are 12 mm. in diameter, and show a hardness of 65 Rockwell.

B. *Test Procedure.*

The four-ball apparatus was said by its inventors to be suitable for evaluating extreme-pressure lubricants. Since extreme pressure agents are supposed to prevent, above all, the seizure or wear of metallic surfaces, they used as critical-value, that load (increased step-wise) which first would produce a sharp and sudden friction-increase "within one minute," immediately followed by a sudden and complete seizure of the metal. We perfected this procedure later, namely, during the one minute the friction-time chart was studied, and from it determining the time from first signs of friction to complete metal seizure. This so-called period of wear or seizure delay in relation to the final load yields a characteristic curve for the particular lubricant under test. The magnitude of the area of wear or wear-values was also determined, based on the final load which gave the seizure.

It should be mentioned that in all tests the load is applied first before starting the motor, which is disengaged later, after the one-minute period is completed.

The several initial tests carried out under identical conditions resulted in wide variations. We assumed that this was in part due to the poor reproducibility of the procedure, especially to conditions present when the motor was first started. By this we mean that the initial load may so have misshapen the true surface of the steel balls that they, variously in each case, were not rotating in true circular fashion at the very beginning of the test. We therefore made a series of tests where the load was applied after starting the motor. The scattered results disappeared and we obtained concordant readings and good checks in all cases. We therefore adopted this procedure in the tests described below.

Inspection of seizure diagrams, (one-minute readings) gave values which were very erratic, allowing no extrapolation of the curves whatsoever. We therefore ran the tests only to point of seizure, using the previously mentioned automatic throw-out clutch which is itself protected from injury by a flexible coupling.

In order to save time we did not pursue the matter of constructing diagrams but noted the time merely from the starting of "first-load" up to initial friction and final seizure. The latter is noted when the automatic disengaging clutch goes into action.

To differentiate the selected test-procedure from methods described elsewhere mention is made that by selecting definite pointers and using certain contact arrangements, the end of the test (definite seizure) can be more clearly indicated. The following items are noted: the appearance of the wear surfaces at the end of a run before seizure takes place shows the presence of a very small (about similar to optical flattening) area of wear. The surface condition of this area shows that it was due to a considerable removal of bearing-metal. The diagrams we constructed, however, showed in those runs which were terminated at the very first instance of friction-increases, a steady deviation of the curve.

Besides the erratic curves, the use of such balls

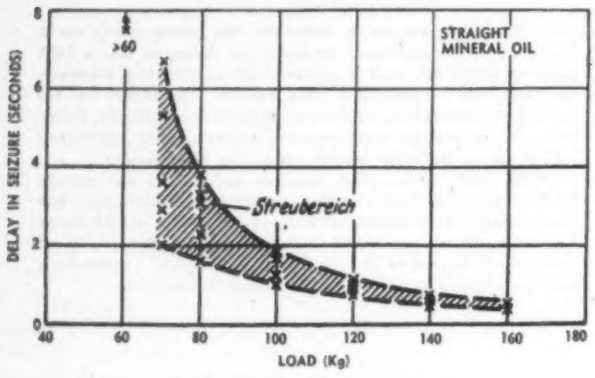


FIGURE 2
Time of seizure-delay related to load.

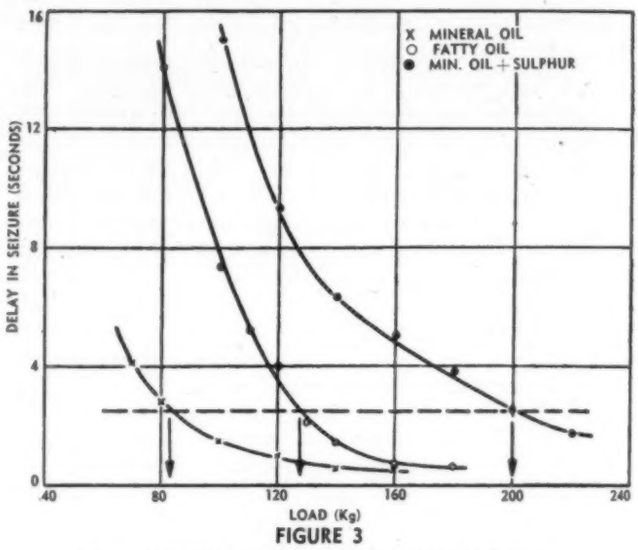


FIGURE 3
Seizure-delay time vs. load for three various lubricants.

gives very indefinite seizure points. The picture points to possible variations in metal make-up or manufacture of the balls themselves, factors which also would determine how they would behave on rotation, etc.

True seizure, then, is the point of initial metallic contact beyond which no normal but only erratic results are obtained. Sometimes this point is passed and then there are cases (when the test is still running) when a sudden decrease in friction takes place. A later and final friction increase will occur and complete seizure finally results. This usually results in loss of temper of the steel balls. By our described procedure we encounter this phenomenon very rarely since we end the run at the first note of steady friction increase. Measurements of the areas of wear is done by measuring the length and width with a microscope.

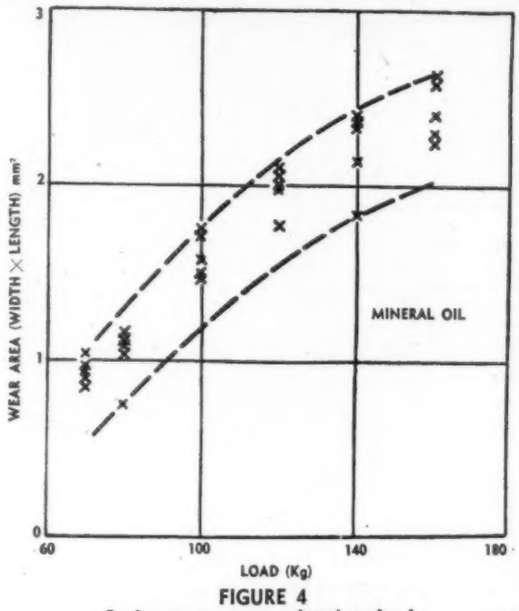


FIGURE 4
Surface-wear areas related to load.

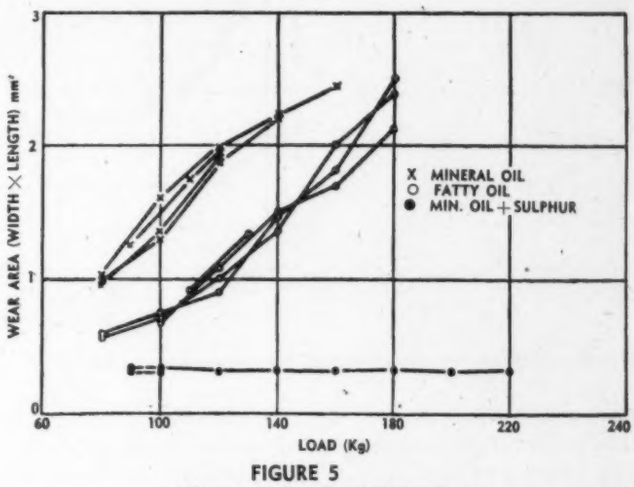


FIGURE 5
Wear tests for three lubricants

C. *Oils Tested.*

We selected two oils containing no oiliness or extreme pressure additives in order to determine what differences, if any, the four-ball tester would reveal. The assumption that a pure mineral oil and a 100 percent pure fatty oil would show extreme differences in friction coefficients led us to select a pure mineral oil (motor type) as a comparison oil (normal oil), and a pure fatty oil of 155.6 saponification value.

Tests made by the I. G. Farben had convinced them that the conditions in the four-ball tester are not to be viewed as those of true bondary lubrication but perhaps more similar to partial lubrication. In this connection viscosity of the test oil enters the picture, and modifies the results to some extent. This type of viscosity influence is fairly well eliminated in the case of the two oils we have chosen, since their viscosities of 18″ F. for the mineral oil and 17.9° F. for the fatty oil, lie very close together.

In order to test out the effect of addition agents in relation to the pure mineral oil, we added sulphur, another lubricant substance, to the mineral oil. In order merely to study the effect of sulphur itself, this substance was ground up in the oil at room temperature, adding the sulphur to the amount of 5 percent on the weight of the oil; this was done rather than first dissolving the sulphur in some fatty oil at higher temperatures. As was to be expected, a considerable portion of the sulphur finally settled out of the mineral oil; the clearer top oil was used for testing without further investigating what percent of sulphur had gone into true solution. A slight increase in viscosity of the blend was taken, as usual, to be a customary occurrence.

Results of Tests

A. *Seizure Prevention (Extreme Pressure).*

Preliminary tests had shown that reproducibility of wear-curves can only be relied on after making many tests. Additional operational precautions and innovations gave no further proof of improving any single reading or result. Tests were therefore conducted from now on in a manner such that at every new increment of load several runs were made at that load in order to obtain a true average of results; in fact, it proved that at least five runs were necessary in order to derive a true arithmetical mean value, and a fairly smooth curve.

Diagram in Figure 2 shows the single values of a series of some five test runs made on the mineral oil. The shaded area between the two curves depicting the limits of scattered results is not always as broad as in this case with our mineral oil but it does show that in the lower brackets of load there exists a region of poor reproducibility.

The average values of runs made on three lubricants mentioned above are graphed in Figure 3.

B. *Wear Values*

The determination of the wear retardation values was carried out by measurement of the depth, width and length of the area of wear on the three stationary balls. The areas in most runs were not circular but mostly eliptical wear spots. Our procedure was not to obtain an average diameter[2] but we employed a unit which is the product of the greatest width times greatest length, and the values averaged for the three balls.

Figure 4 shows the results for the mineral oil. Despite a somewhat scattering of the area of true values, the graph shows smooth curves can be obtained as the result of five test runs.

In Figure 5 are shown the results of all three oils, the mineral oil, fatty oil and the sulphurized oil. The points of several runs are connected.

For all oils tested there seems to be a relation between wear and load as well as between seizure and load; therefore, a relationship must also exist between wear and seizure. This is depicted in Figure 6.

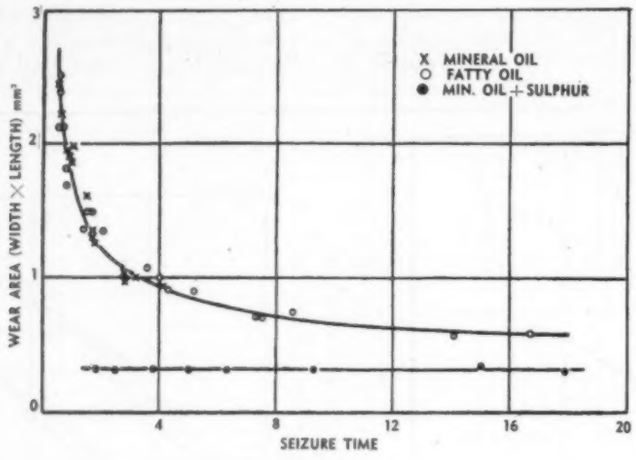


FIGURE 6

Wear tests vs. seizure time.

Meaning Of Results

From Figure 3 we noted that tests on the three lubricants gave clearly quite different results when using the four-ball apparatus. For instance, the fatty oil shows good extreme pressure value in comparison with the straight mineral oil. Unfortunately the differences become less apparent at higher loads, and just where reproducibility is quite good. The sulphurized oil, however, even at very high loads is very much better than the mineral oil in all tests—probably some tenfold improvement.

To evaluate many hundreds of curves, Boerlage and co-workers selected the so-called 2.5-second seizure period. From Figure 3 it can be seen that this selection is of value, since in this 2.5-second interval great differences occur, though they are still quite reproducible.

The results of the four-ball test by our procedure is as follows: (See arrows in Figure 3):

Straight mineral oil	83 Kgm. seizure load
Fatty oil	128 Kgm. seizure load
Sulphurized oil	200 Kgm. seizure load

The mineral oil had previously shown a value of 78 Kgm., when tested at the Delft laboratory using the 2.5-second time-period limit. They used, however, one-half-inch steel balls, and perhaps some variations in test procedure of which we are apparently unaware.

The curves in Figure 5 are very interesting as to retarding seizure. Wear curves, however, do not seem to be as conclusive as the former. Of course, the mineral and fatty oil do behave alike in that they do appear to cause final wear with increased loads; in contradistinction to these two oils we note that the sulphurized oil behaves quite differently—an almost constant wear value is obtained eventually regardless of increasing load.

Figure 6 depicts the situation more clearly how sulphur addition changes the wear picture as we obtain it in the case of the straight mineral oil. Slopes of curves for the former are constant, and if low magnitude, those for the mineral oil, at low loads and therefore higher wear retardation, show greater slopes and curvature of the lines.

It is quite apparent that values for the fatty oil lie actually on the same curve as the mineral oil. All mineral oils with or without fatty-oil additions were found to graph the same way, regardless of origin or type of fat. These oils yield a definite curvature at some definite time limit of seizure prevention and anti-wear.

For certain load ranges the wear values lie on the lower flat part of the curve in the case of good oils; with oils showing poorer anti-wear value, or less valuable oils, the wear value versus load is shifted to the left or steep part of the curve, where wear-values are high.

Small or very valuable wear-values as shown by the sulphurized oil must be attributed to an entirely different set of conditions and forces than are present in mineral oil-fatty oil lubrication. In the case of the sulphur oil we noted that the steel balls took on a dull black polished appearance—perhaps iron sulphide layers—especially at the very edges of the wear area; mineral and fatty oils give no such coatings.

Especially important is to take care that all balls be of the same make and same dimensions when running many tests, otherwise no concordant results are possible.

Continuation of researches in this direction should give valuable new information of the behavior of films and their value in lubrication. Results obtained by the four-ball machine need still further study and the values need further practical correlation.

LITERATURE CITED

[1] G. D. Boerlage, "The Four-Ball Tester for E.P. Lubricants," Engineering 136, 14 (July) 1933.

[2] H. Blok, "Seizure Delay Method for Determining the Seizure Protection of E.P. Lubricants," S.A.E. Jour. 44, 193 (1939).

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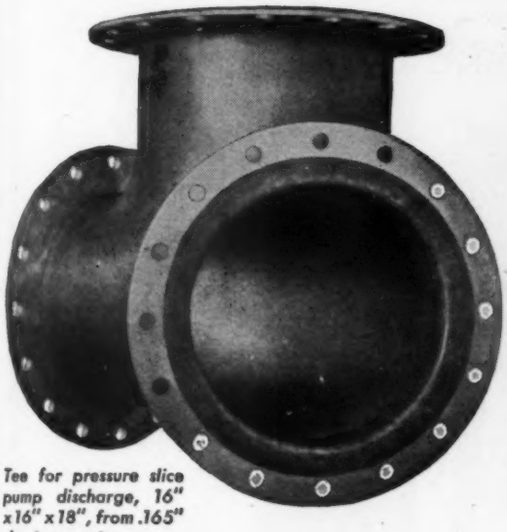
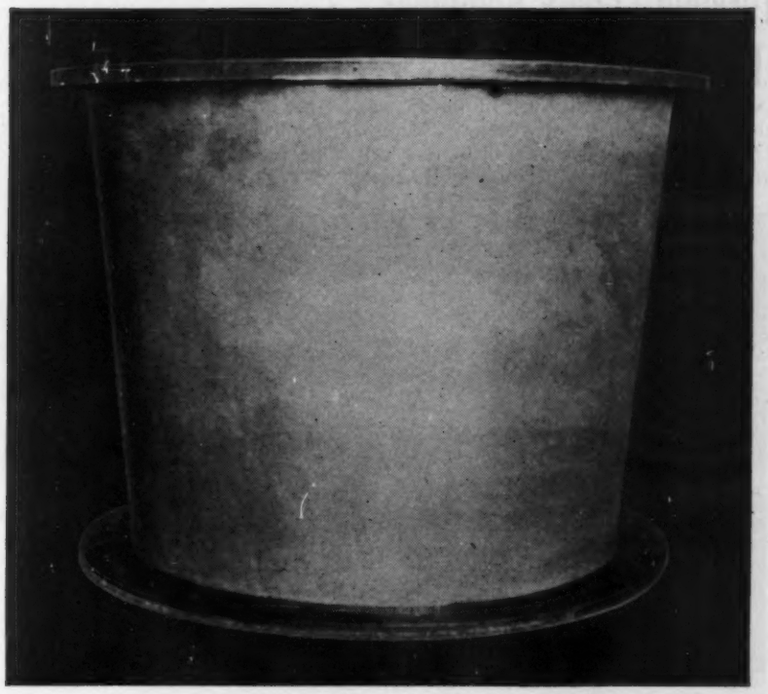


Tee for pressure slice pump discharge, 16" x16" x18", from .165" sheet copper



Tapering evaporator connection, 42" to 36" diameter, from .250" sheet copper

Steam belt for evaporator, 9' 1.25" diameter x 42", from one sheet of 5-16" copper. Contains Muntz Metal head ½" thick, and 800 copper tubes. Finished weight, 17,000 lbs.

Special elbow, 18" x 14" x 18", made from .165" sheet copper

Flanged elbow for paper stock, 24" diameter, from .165" sheet copper

This equipment was fabricated of Revere sheet copper by R. A. Burroughs & Co., Inc., Norfolk, Va. All seams were gas welded with copper welding rod.

THE pictures on this page show plant equipment in unusual shapes and large sizes, fabricated of Revere sheet copper by R. A. Burroughs & Co., Inc., Norfolk, Virginia, coppersmiths and metal fabricators since 1910. They illustrate the amazing versatility of this metal, which in skilled hands can be worked from the flat into practically any shape.

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OPERATION HOW TO DO IT MAINTENANCE

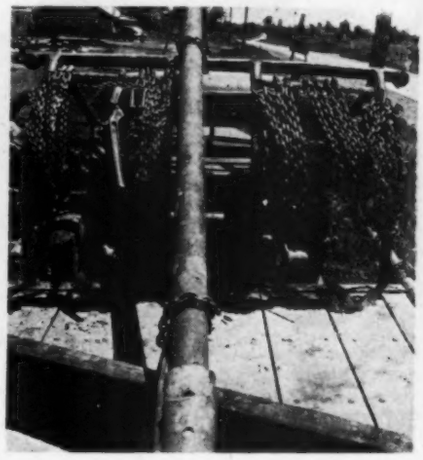
Artificial Eye Poster Is Forcible Goggle Reminder

TO FORCIBLY bring to the attention of mechanics using grinding wheels the necessity of wearing goggles, Union Oil Company places artificial eye advertising on a stand above the starting button. The placard informs the user that he may obtain glass or plastic eyes from a certain manufacturer. Men approaching the grinding wheels with chisels or other tools requiring shaping on the wheels take time to place goggles over their eyes before starting the motor. In several instances men took the goggles to the facilities providing for cleaning so that clear lense would be in shape for use. No one has used the wheels without goggles.

Artificial-eye advertisement is effective goggle warning

Truck-Cab Guard Makes Place for Boomer Chains

THE rack commonly placed just behind the cab of a truck used for hauling pipe and other heavy refinery equipment usually is utilized as support for spare boomer chains and other load binders. Unless anchored, these chains sway with truck motion, pounding against the cab and causing so much noise that it is sometimes difficult to check other operation noises of the equipment.



Truck Cab Guard

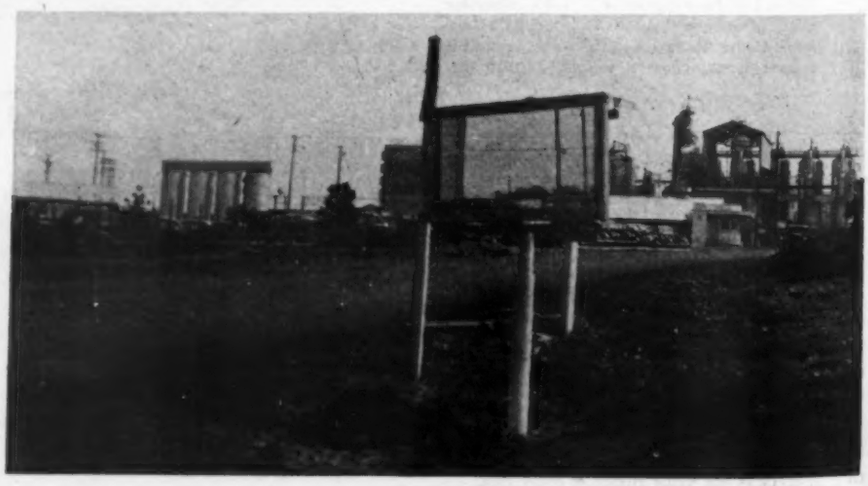
By welding expanded-metal mesh across this cab guard, one maintenance man forms a support for the chains which keeps them clear of the cab, and prevents their possible fouling the truck winch as it is being used to handle heavy equipment onto the truck bed.

The sloping support also aids in hosing off the chains when they have been used to extricate the truck or other equipment from a mudhole, and also

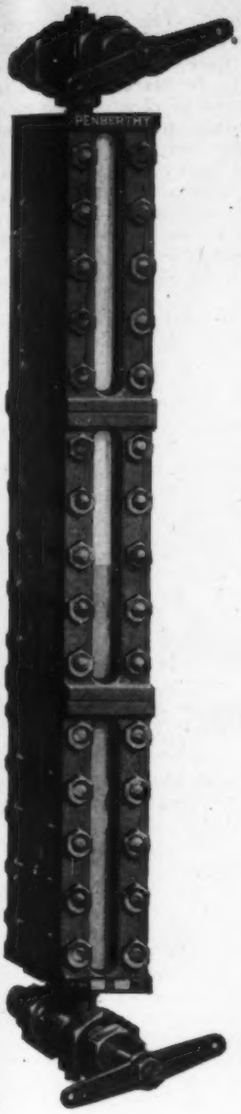
Mirror Promotes Safety At Parking Lot Exit

A LARGE plate-glass mirror at the exit of the refinery parking lot assists in reduction of traffic mishaps. The mirror shows traffic on the busy highway.

Drivers making a left turn into traffic from the lot can see the road into which they are turning, but not so readily the cars coming from the other direction. A 24 x 36-inch mirror mounted as shown in the photograph is in effect a rear-view mirror which shows traffic approaching from the right. As adjusted, this mirror provides a view of the highway for approximately 1000 feet. This idea is one worthy of public traffic-safety officials.



Rear-view mirror for parking lot



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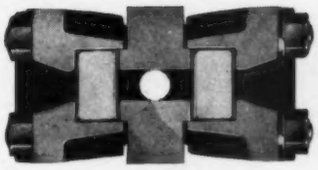

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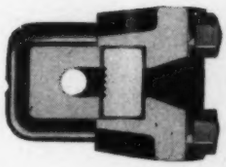

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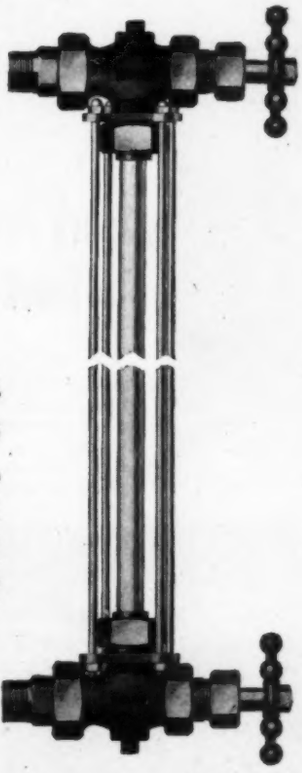
DROP FORGED STEEL LIQUID LEVEL GAGES

Liquid shows black — empty space shows white. Preferred wherever liquid level must be easily and positively visible . . . and when liquids are under high pressure or at high temperature.



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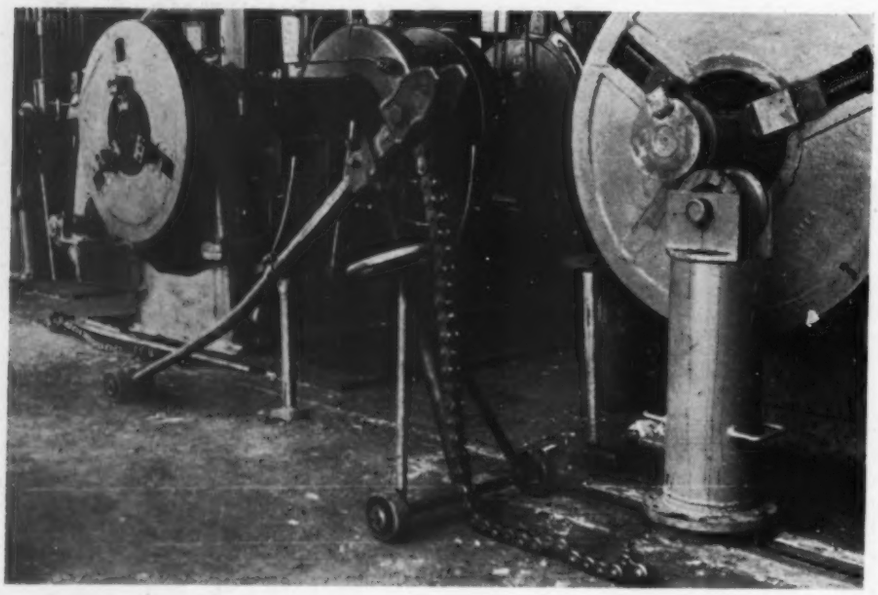
makes easier visual examination of the links for possible damage.

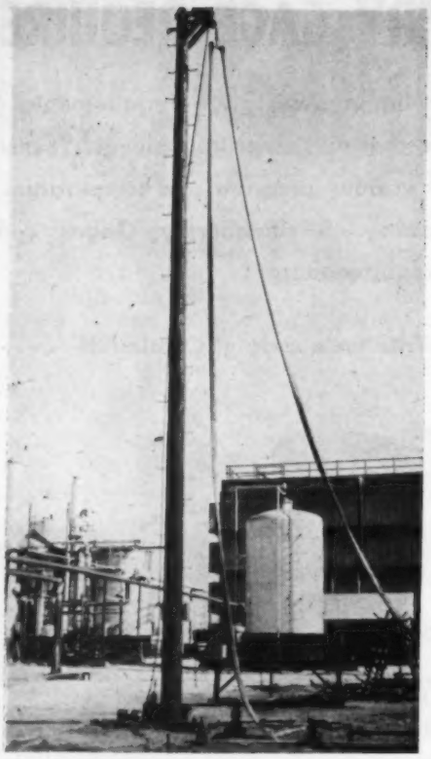
Since the mesh does not extend out farther than the original support on which it is mounted, it does not interfere with normal loading of the truck.

Dolly Helps in Using Heavy Pipe Tongs

WHEN breaking out fittings on salvaged pipe, the job of frequent fitting of the heavy tong back-up soon becomes arduous. To eliminate the heavy work, a mechanic in Union Oil Company's Oleum refinery shops devised an attachment for the chain tongs that eliminated all of the heavy lifting. He attaches a pair of small trucks to the point of the chain tongs for easy rolling along the floor. Near the other end is placed a substantial clamp holding a long threaded stem passing through a wheel from a gate valve. Another pair of trucks with a tube receiving the threaded stem support the jaw end of the tongs. Adjusting the height of the back-ups is done by spinning the wheel up or down. To support the point end of the tongs, is a standard with a pad which can be run up or down to suit the load and conditions of work.



Pipe tongs mounted on wheels.



To dry fire hose

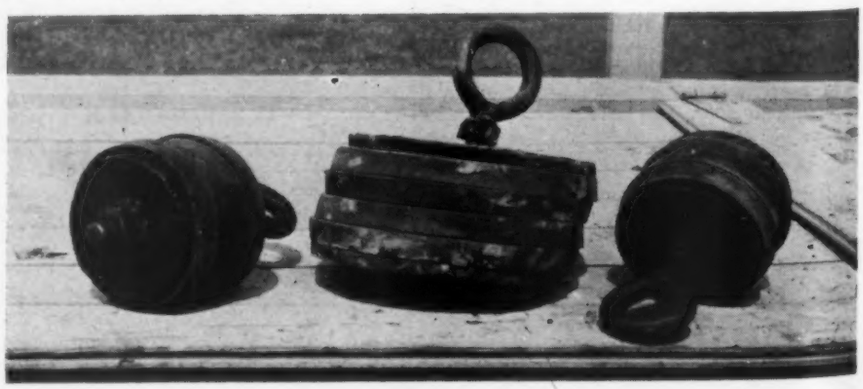
Removable Line Plugs Expanded Inside Pipe

FREQUENTLY it is desired to test lines without arranging for tying in a valve or making a weld to close the open end. If the open end of the line is such that a sleeve cannot readily be placed, and the blind in the sleeve braced against endwise travel under pressure, the closure must be placed within the line.

One company has developed a pressure-set plug which will withstand normal test pressures, and which is readily placed or removed. The closure consists of a heavy steel plate washer, slightly smaller than the inside diameter of the line to be tested. A hole is bored at its center to clear a one-inch bolt, and a one-inch nut is welded on the outside, aligned with the hole. Two or more pairs of heavy elastic washers, of rubber or oil-resistant synthetics, are placed on the plate washer. The pairs are assembled with the bevels facing in opposite directions, to utilize the pressure of any leakage as further sealing effort.

A second steel plate completes the pressure portion of the plug, the two being forced against the gaskets through the one-inch eye bolt. This unit carries a second nut, welded to the shank so as to bear against the second backing plate.

Friction of the inner plate against the sealing material prevents turning of the nut, and allows the material between the plates to be expanded as much as desired, so as to build up pressure against the pipe wall.



Removable line plugs

Hose Suspended on Pole Insures Proper Drying

THE life of fabric fire hose depends upon its condition when spooled after use. Moisture breaks down the cotton fibres and rapid deterioration sets in. But dry lengths last for many years, and to condition the hose after use, drying is assured when hung in the air and sunlight. To accomplish rapid drying, a bracket is attached to the top of a lamp pole with a sheave hooked in an eye bolt for a manila or wire line with a loop for connecting a bar with apparatus for attaching the female ends of the fire hose. A counterweight on the other end of the line assists in raising the hose when elevated for drying.

Spark Arrestors Put On As Trucks Enter Yard

TO PREVENT refinery fires due to carbon particles leaving truck exhaust pipes, Union Oil Company provided spark arrestors which are fashioned to fit the size of any exhaust commonly used on diesel motor vehicles. The spark-arresting part of the apparatus

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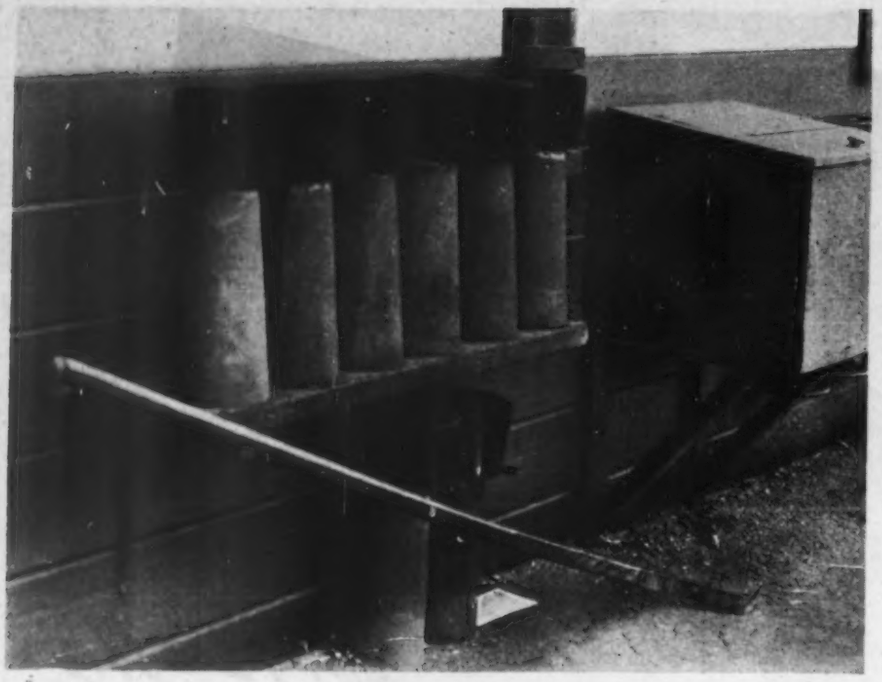


Spark arrestors for diesel truck exhausts

consists of a drum shaped section made of heavy woven wire cloth. This is attached to a funnel-shaped cylinder which fits over the exhaust pipe. Lugs with holes are attached to opposite sides of the cones at the point where the screens are attached so that the gateman may lift the arrestor with a pole fitted with a fork that fits into the holes in the lugs. These fittings are placed on the vertical exhaust of the diesel transports at the refinery gate, and removed when the vehicle leaves the yard.

Blanking-Off Plates Always at Hand

WHEN a pipe-line manifold is constructed to fit each individual flange connection, it is difficult to insert blanking-off plates without distorting the fittings when drawing up flange bolts. Plates with companion ring plates for each connection which may be shut off during repairs is the answer. This apparatus consists of the two members; the blank plate to shut off the line, and the companion ring for use when the line is in service.

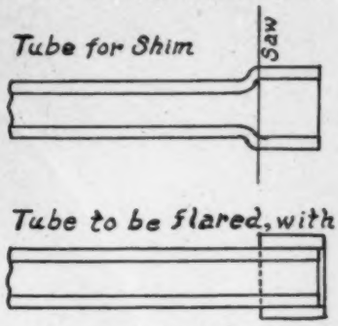
The blanking-off plates are cut from substantial steel leaving tabs on opposite sides for convenience when handling and to provide means of connecting the plate to the companion ring. The ring is shaped in the same manner except that the center is cut away to the same inside diameter as the flange where it is to be installed.

When the line or valve is in service the companion ring is installed between the bolted flanges with gaskets on each side, to occupy the space taken up by the blank plate when it is inserted. This method is employed so that the companion flanges are always the same distance apart. The ring spacer and the blanking-off plate are chained to each other so that both are always at the point where needed.

Safe waste flume

Shop-Made Shim For Retubing Old Condenser

MANY old condensers, which have a header plate that has deteriorated to such an extent that rolling in new tubes is impossible, may be repaired satisfactorily with only the equipment necessary for an ordinary replacement.

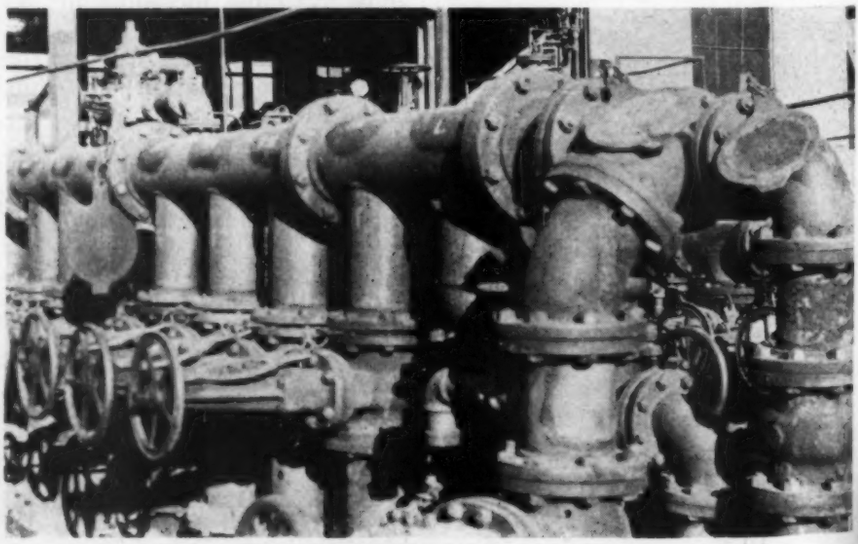


A new tube is placed in a vise and flared to the maximum capacity of the roller. This flared section is then removed, with a hacksaw, just below the enlarged portion. This slightly tapered sleeve is then driven on over the end of a new tube, forming a thick shim. The tube may then be rolled in, forming a tight fit in the corrosion-enlarged hole in the header plate.

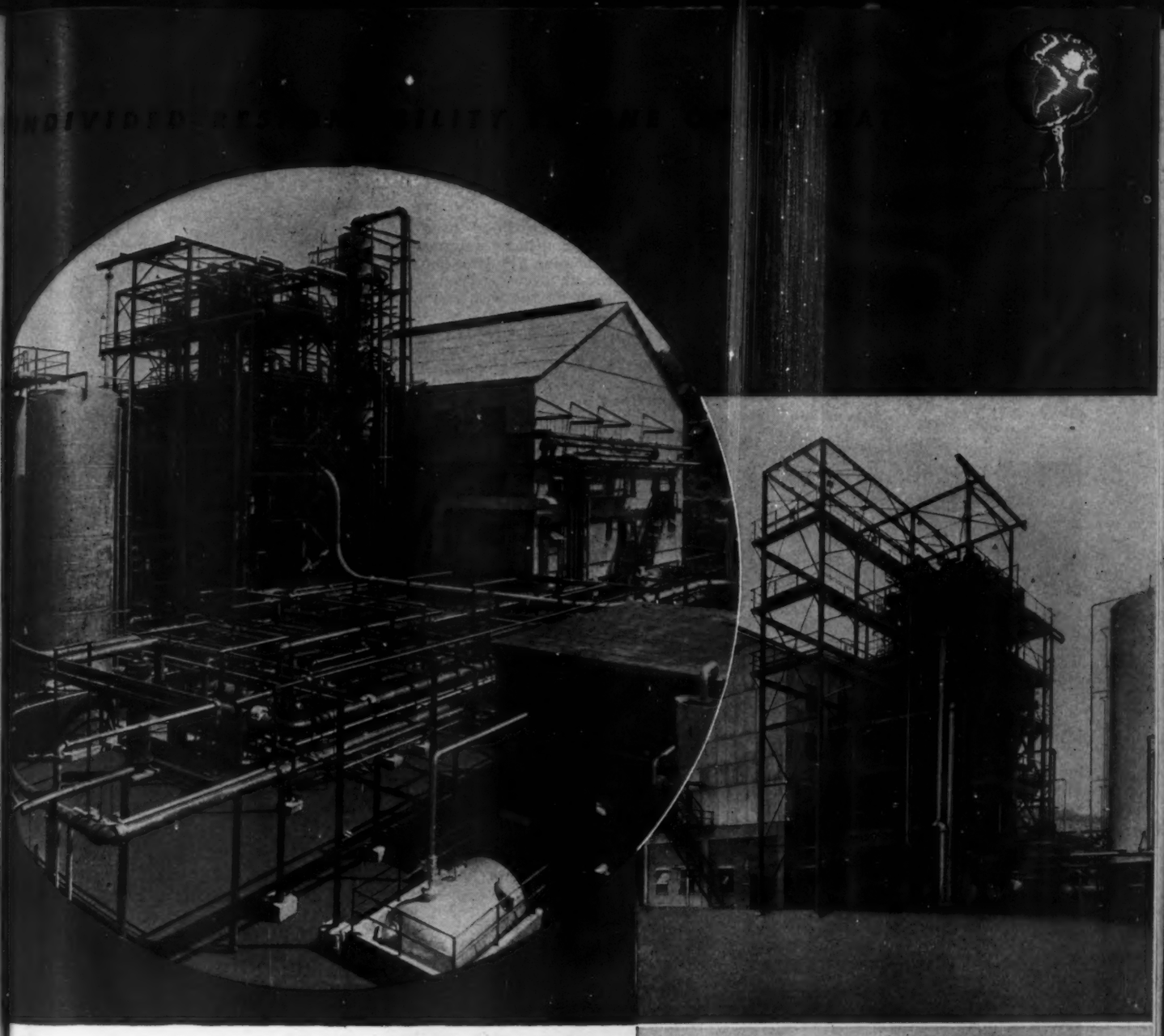
Safely Covered Waste Flume Provides Walkway

WASTE material, flushed at intervals from stock tanks and process equipment, is usually inflammable and thus poses a fire hazard when carried around the plant in open ditches. Flumes and other open carriers also tend to fill with wind-borne trash, dust and dirt and, if the waste products adhere to the sides of the ditch, eventually clog the run so that thorough cleaning is required.

One refinery solves its waste-disposal problem by erecting a sheet iron flume, with good hydraulic gradient, extending

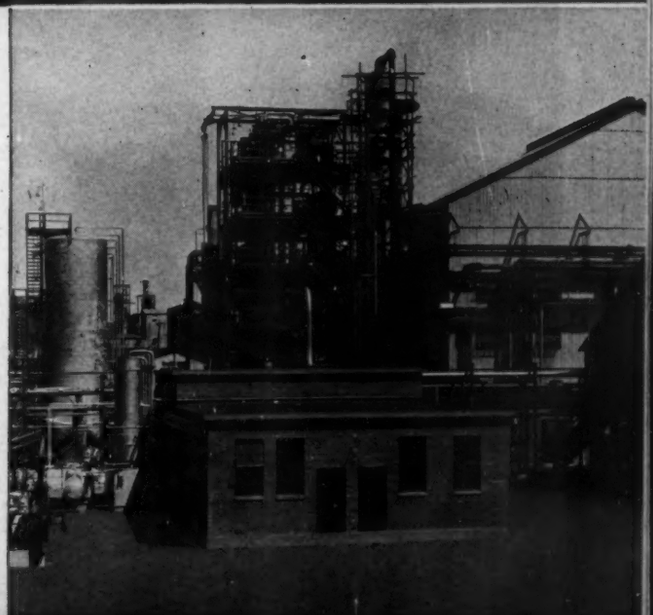


Blanking-off plates for flanged connections



M·E·K SOLVENT DEWAXING PLANT

The photos on this page show three views of a 2000-barrel M·E·K Solvent Dewaxing Plant recently designed, engineered and constructed by Arthur G. McKee & Company under a Texaco Development Corporation process license.



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Adjustable forge hood

Adjustable Hood Handles Forge Smoke Nuisance

AN ADJUSTABLE hood over the blacksmith's forge insures removal of fumes from the building even when the initial fire with its excess of smoke is started, and is out of the way when the fire is being used.

The hood here discussed was built by a tinner. It is attached to a permanent stack through the roof. The adjustable feature consists of a sleeve fitted over the lower end of the stack, which carries the hood covering the forge. Pulley and cables attached to the movable section and to a band around the permanent stack carry counterweights to permit easy adjustment of the hood. A latch to clamp the hood in full up position is manipulated by a cord.

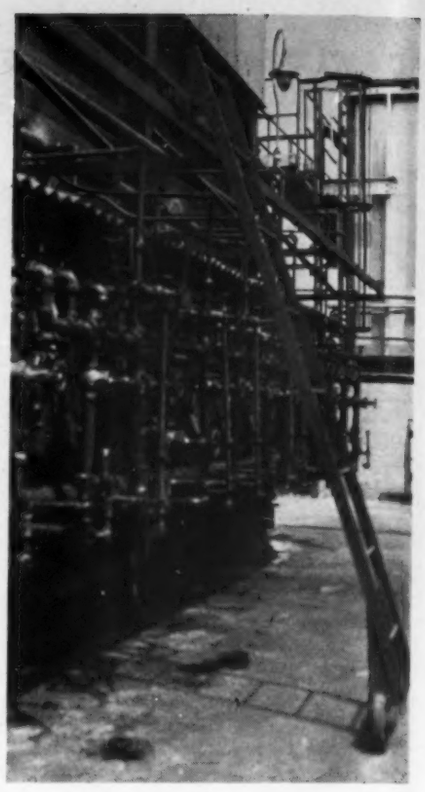
When building the fire in the morning the hood is drawn down to within 15 inches of the fire so that the smoke goes up the stack. When the fire clears and is in use the draft induced by the heat removes the normal fumes through the hood in its elevated position.

from the tank battery to the burning pit. A close fitting cover is provided, bolted at intervals to the flume and forming, in addition to a cover, a convenient walkway from battery to pit. Safety tread steel plate is used, so as to facilitate the use of the cover as walkway.

The flume is brought into the waste pit through the firewall, and is provided with screened stops which are placed before the pit is ignited. This prevents flashback of flames from the pit through the flume, and safeguards the stock tank battery.

Traveling Ladder Aids Fireman in His Work

A CONVENIENCE for firemen may be obtained by installing a ladder on wheels to be moved back and forth in front of the unit so that the fireman can make adjustments on valves and controls above normal reach. This ladder is all steel, with rubber-tired wheels. The upper end is equipped with metal grooved wheels which rest on a track above the burner controls of the heater. The track is firmly attached to the structural steel of the heater and extends outward so the ladder may be moved from one end of the unit to the other without difficulty.



Traveling ladder for fireman

Expansion-Joint guard

Expansion Joint Travel Limited by Chain Guard

EXPANSION joints, required where steam or flow-lines carrying hot products are laid in long tangents, are of two general types: the bellows or expanding diaphragm types, which is effective only if the thermal shift is not excessive; and the sleeve type, which relies on packing to maintain a seal, and whose lateral change is limited only by the length of sliding or telescoping portions of the unit.

Since the sleeve type depends upon free action between parts to relieve expansion stresses, some type of anchor is required to prevent the pressure within the line acting to force the slip joint open. Where the runs are long, and end anchors are not available, one plant welds heavy ears to the line pipe on each side of the expansion joint, passing a heavy chain through the eyes of the ears, and hooking the ends together with sufficient slack to provide for expansion shifts, but snubbing travel before the joint can travel endwise to the point where the telescoping parts no longer engage.

Where the line must be lagged for heat conservation, the two ears for attaching the chain are made long enough so that the links clear the lagging. The packing of the joint may be serviced or replaced without removing the chain.

THE MONTH IN THE INDUSTRY

Carthage Hydrocol Elects Gabrielson President

G. G. Gabrielson has been elected president of Carthage Hydrocol, Inc., recently organized with plans for construction of a plant in the vicinity of Brownsville, Texas, for synthesis of natural gas. Gabrielson has been general counsel for the company, and will continue in that position as well as in general law practice in New York and New Jersey.

Carthage Hydrocol, Inc., is capitalized at $19,000,000, of which $10,000,000 is supplied by eight companies, including The Texas Company, Forest Oil Corporation, Niagara Share Corporation, United Gas Corporation, La Gloria Corporation, Stone & Webster, Inc., Gulf States Oil Company and Chicago Corporation. Reconstruction Finance Corporation has agreed to loan an additional $9,000,000. Directors of the company include George H. Clifford, president of Stone & Webster Service Corporation; Richard J. Dearborn, president of Texaco Development Corporation; Michael Halpern, vice president of The Texas Company; Percy C. Keith, president of Hydrocarbon Research, Inc.; Harry T. Klein, president of The Texas Company; John F. Lynch, vice president of La Gloria Corporation; H. Neil Mallon, president of Dresser Industries, Inc.; W. Frank Miller, vice president of Forest Oil Corporation; J. Fred Schoelkopf IV, president of Niagara Share Corporation, and Gabrielson.

Announced plans for the plant involve an expenditure of $15,000,000. Gasoline and other chemical components will be synthesized from gas by a process developed by Keith and his associates in Hydrocarbon Research, Inc.

G. G. GABRIELSON

Conventions

September

16-20—National Instrumentation Conference and Exhibit, Wm. Penn Hotel, Pittsburgh.

18-20—National Petroleum Association, Hotel Traymore, Atlantic City.

October

3-5—American Institute of Mining and Metallurgical Engineers, Petroleum Division, Galveston, Texas.

7-9—American Society of Mechanical Engineers, National Conference, Tulsa.

11—California Natural Gasoline Association, Annual Fall Meeting, Ambassador Hotel, Los Angeles.

7-12—American Gas Association, Atlantic City.

November

7-8—Society of Automotive Engineers, Fuels and Lubricants Meeting, Tulsa.

11-14—American Petroleum Institute, annual meeting, Stevens Hotel, Chicago.

CGNA to Hold Annual Meeting October 11

The annual meeting of the California Natural Gasoline Association will be held October 11 in the Ambassador hotel, Los Angeles. Morning and afternoon sessions will consist of presentation of technical papers, followed by a dinner and entertainment in the evening.

The program is being worked out by a committee headed by E. G. Ragatz, Bectol Brothers McCone Company. A recent news release from the office of George Tyler, secretary, gave the following summary:

Opening the morning session, Dr. B. H. Sage, California Institute of Technology, will present data and methods which natural - gasoline - plant designers and process engineers can use to accurately predict the thermodynamic properties of hydrocarbons. Then C. D. Gard and Hugh Colvin of Union Oil Company will discuss the economics of LPG operations from the manufacturer's viewpoint. The final paper of the morning session will evaluate the various methods available for processing absorption-plant rich oil.

In the afternoon, following. President Kibre's address, Frank West of the engineering firm of West and Cole, will discuss the selection of absorption pressures for plants to be installed in high-pressure fields. In the second paper, R. C. Alden of Phillips Petroleum Company will discuss natural-gas conversion by the Fischer-Tropsch process. The final paper of the meeting will be presented by Ragatz and will deal with the determination of absorber operating efficienies.

The evening session will be in charge of Entermtainment Chairman P. W. Mettling of Cooper-Bessemer Company. Mettling stated that his committee is rapidly completing arrangements for the dinner and show and that both would be of prewar quality.

Baton Rouge Facilities Are Being Expanded

Along with the announcement that the Baton Rouge plant of Standard Oil Company of New Jersey had processed its billionth barrel of crude oil, M. W. Boyer, general manager, outlined improvements and additions now under way and in prospect for the Louisiana plant. The plant is the first within the New Jersey group to reach a billion barrels total throughput.

A new synthetic alcohol unit is under construction at Baton Rouge, as well as replacement of an old section of the docks. Work has begun on an expansion of the research laboratory facilities.

Engineering designs are under way for an expansion of gasoline manufacturing facilities as well as added capacity for the production of wax. Some of the construction represents work held over during the war period.

The Pure Oil Company Plans New Laboratory

The Pure Oil Company has announced tentative plans for construction of a new oil research and development laboratory at Crystal Lake, Illinois. Negotiations are underway for purchase of a 68-acre site upon which buildings and facilities of $400,000 initial cost are contemplated. The company's laboratory now is located at Northfield, Illinois.

Dinkins Made President Jefferson Chemical Co.

P. M. Dinkins has been made president and a director of Jefferson Chemical Company.

This company was organized in 1944 by American Cyanamid Company and The Texas Company to produce chemicals from petroleum and petroleum

P. M. DINKINS

CALIFORNIA'S LARGEST CYCLING PLANT USES

9 Clark Compressor

South Cole's Levee Unit Cycling Plant, Ohio Oil Co., ...tor, gas is returned to the sand at the rate of 45,000,000 ... per day under 3,700 lbs. pressure.

...e Clark 600 BHP Right Angle gas-engine-driven units ...rise the compressor installation in this modern plant, ...h is located on Buena Vista Lake at the base of Elk Hills, ...rnia. Products manufactured are condensate, propane, ...tane, normal butane and natural gasoline.

...RK BROS. CO., INC., Olean, New York

...ORK • TULSA • HOUSTON • CHICAGO • BOSTON • LOS ANGELES
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Consider These Clark "Angles"

1. Clark 2-cycle economy and dependability, proved repeatedly in many types of plants, under most exacting conditions.

2. "Custom-tailored" to fit precisely the type of processing and special requirements involved.

3. Comprehensive engineering service from inception of the project to operation in the field.

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...E OF THE DRESSER INDUSTRIES

SETS THE PACE IN COMPRESSOR PROGRESS

THE MONTH . . .

gases. Its first plant, now under construction at Port Neches, (Beaumont-Port Arthur), Texas, will produce intermediate chemicals used in the synthetic rubber, plastics, textile, and other industries.

Dinkins has been associated with American Cyanamid Company since 1923. He received his B. S. in chemical engineering from Massachusetts Institute of Technology in 1918.

Monsanto to Add Plastics Unit at Texas City

Mosanto Chemical Company, St. Louis, will add plastics-making facilities to its Texas City, Texas, styrene plant recently bought from the government. The styrene plant, which the company operated during the war, will continue to provide styrene for the synthetic rubber program.

The styrene unit also will provide raw material for the polystyrene plastics unit to be added at Texas City. Polystyrene will be furnished to manufacturers of finished products such as combs and dresser sets.

California Refining Company Starts East Coast Operations

California Refining Company has been organized under the terms of an agreement between Barber Asphalt Company and Standard Oil Company of California. The agreement is awaiting approval of stockholders of Barber Asphalt Company.

The new company will operate the refining plant, marine terminal and other facilities near Perth Amboy, New Jersey. Crude oil produced in Louisiana by The California Company, subsidiary of Standard Oil Company of California, will be processed at the New Jersey plant, whose present capacity is 15,000 barrels daily.

Fred Powell, who has been manager of the refining division of the El Segundo plant of Standard Oil Company of California, has been named president of the new company. He has been with the company since 1925 when he went to work in the Richmond plant. Subsequently he held positions in the company's plant at El Paso and Bakersfield, going to the El Segundo plant in 1941. Powell is a graduate of the University of Washington.

FRED POWELL

Standard of California Announces Promotions

C. A. Pollard has been appointed assistant general manager of Standard Oil Company of California's Richmond refinery. Formerly manager of the refining divisions at this refinery, Pollard has been with the company since 1921. Succeeding him as manager of the refining divisions is C. W. Rehfuss, who formerly was manager of the service divisions at Richmond. Rehfuss has been with the company for 22 years.

Dr. P. C. White Appointed Whiting Chief Chemist

Dr. P. C. White has been appointed chief chemist at the Whiting plant of Standard Oil Company (Indiana). His promotion from assistant chief chemist fills the vacancy caused by the retirement September 1 of F. M. Rogers. Dr. White joined the organization in 1938 after finishing his graduate work at the University of Chicago.

R. F. Marschner has been made section leader in charge of exploratory research on petroleum processes. Dr. Marschner received his bachelor's degree from Brown University and his Ph.D. from Penn State. He has been with the company since 1934.

Other members of the research staff have been promoted to the following positions: J. C. Bailie, acting assistant chief chemist; W. J. Tancig, group leader in charge of analytical methods application; A. P. Lien, group leader in exploratory research; J. J. Lukes, group leader in the pilot plant section; and H. S. Wood, group leader in the light oils section of the technical service division.

Dr. Rogers, retired after 38 years' service with the company. A pioneer oil chemist, his most important contribution was perhaps the part he played in developing the Burton process, and in solving the problems involved in refining the cracked products. At a dinner in his honor, Dr. Rogers, was presented a set of matching luggage by J. K. Roberts, general manager of research, on behalf of his associates. Dr. R. E. Humphreys, retired vice president in charge of manufacturing and chief chemist when Dr. Rogers joined the company, was principal speaker at the dinner.

Smith Foreign Refining Coordinator for Standard

L. G. Smith has joined the foreign refining coordination department of Standard Oil Company (New Jersey), and has resigned as president and general manager of Lago Oil & Transport Company, an affiliate operating at Aruba, Netherlands West Indies. John J. Horigan has succeeded Smith at Aruba.

Smith graduated from University of Illinois with a degree in mechanical engineering, and has been in the oil business since 1913. Horigan is a Yale civil engineering graduate, and entered the oil business in 1920. He has been a director of Lago Oil & Transport Company since 1942.

Jersey Standard Expands Executive Development

Expansion of the executive development program of Standard Oil Company (New Jersey) and appointment of an advisor to coordinate the work of training future leaders for the business have been announced by Eugene Holman, president.

Holman said the program is intended to develop a reserve of executives of broad training and experience for Standard Oil Company (New Jersey) and affiliated companies.

"The mass production and distribution methods necessary to satisfy the needs of people everywhere result in organizations which in our opinion can best be administered by a decentralized

Trends of Operations and Changes in Stocks

Figures on crude stocks are from Bureau of Mines weekly reports; all others from American Petroleum Institute weekly reports, which are estimates on Bureau of Mines basis.

(All figures in thousands of barrels—add 000)

	Crude Oil			Gasoline		Gasoil and Distillate		Residual Fuel	
Trends in Week Ended:	Production Daily	Runs to Stills Daily	Stocks Week End	Production Weekly	Stocks Week End	Production Weekly	Stocks Week End	Production Weekly	Stocks Week End
1945:									
January 27	4,727	4,756	221,310	14,957	88,223	4,843	33,561	9,252	51,119
February 24	4,777	4,803	219,351	15,500	95,972	4,958	28,753	0,084	46,713
March 31	4,781	4,677	223,782	14,644	98,758	4,548	26,889	9,184	41,745
April 28	4,805	4,780	223,474	14,633	94,068	4,636	28,273	9,379	39,813
May 26	4,887	4,950	222,831	15,194	89,121	4,667	29,184	9,670	38,548
June 30	4,903	4,999	220,781	15,546	86,472	4,910	32,213	9,077	40,488
July 28	4,930	4,996	218,507	16,106	86,008	4,598	36,071	9,586	42,283
August 25	4,892	4,931	211,813	15,986	84,693	4,960	39,782	9,356	46,201
September 29	4,357	3,812	222,387	11,913	79,552	3,940	43,689	7,047	46,853
October 27	4,273	4,838	224,230	15,530	74,335	5,159	43,472	8,691	45,943
November 24	4,469	4,648	219,363	15,681	83,184	4,802	45,258	8,800	47,474
December 29	4,474	4,729	218,918	14,546	95,205	5,055	36,651	8,765	42,447
1946:									
January 26	4,626	4,553	220,544	13,622	107,737	5,720	29,498	8,411	39,722
March 2	4,726	4,779	229,430	13,871	104,462	5,888	25,148	8,634	38,441
March 30	4,425	4,684	221,214	13,896	104,715	5,357	28,240	8,738	37,746
April 27	4,672	4,685	221,689	14,228	99,631	5,568	30,466	9,204	39,404
May 25	4,759	4,857	222,214	14,322	95,769	5,463	32,973	8,908	43,368
June 29	4,957	4,854	223,883	14,500	92,333	5,408	37,762	8,828	46,447
July 27	4,926	4,896	222,740	14,535	88,626	5,817	44,316	8,217	49,517
August 24	4,836	4,866	225,500	14,639	86,251	5,649	51,405	8,126	52,061

CAST NICKEL STEEL

MAINTAINS TOUGHNESS . . . PROMOTES SAFETY

Retention of toughness at low temperatures may be secured in units, large or small, by use of alloyed cast steel. The 600 pound Crane valve shown above is an example, and the table below gives the mechanical properties of the metal.

Here's a metal for applications at extremely low temperature.

At minus 150° F. . . . test pieces sectioned from representative castings have shown an inherent toughness exceeding 15 foot-pounds Charpy impact resistance.

Basically an "ingot iron" of extremely low carbon content, fortified with 3½ to 4 per cent Nickel, this alloyed cast steel is produced by Crane Co. for valves and fittings exposed to low temperature service in a wide range of industries.

Consultation on problems involving the production, treatment or application of Nickel alloys is invited.

MINIMUM PHYSICAL PROPERTIES

Tensile strength	65000 p.s.i.
Yield Point	40000 p.s.i.
Elongation	25%
Reduction of area	45%
Charpy Impact Resistance	
at room temperature	30 ft.-lbs.
at −175 F., −115 C.	15 ft.-lbs.

THE INTERNATIONAL NICKEL COMPANY, INC. 67 Wall Street New York 5, N. Y.

form of management," he said. "This type of administration increases the demand and opportunity for larger numbers of skilled executives than ever before. We believe it is one of the first duties of business to develop leaders of promise and provide the training for such executives."

Under the program, executive training is based on a continuing analysis of executive functions and personnel by committees. These groups have full responsibility for the success of their activities in selecting personnel of the greatest promise for development. After careful appraisals of the qualifications of selected personnel, steps are taken to fill gaps in experience. The company is also sending men to educational institutions for special courses, particularly in general business and management methods.

Managements are encouraged to appoint more than one assistant to department heads as a means of providing an insight into the operation of different branches of the business. Although recognizing that this practice sometimes results in over-staffing the departments from the strict point of view of the efficiency expert, the cost is considered justified when viewed in terms of the long-run benefit.

Appointed to activate the program is George B. Corless, of Houston, veteran oilman, geologist and mining engineer who recently resigned from Humble Oil & Refining Company. He will maintain liaison with affiliated companies in exchanging techniques in carrying on the executive training program. Corless is a graduate of the University of Michigan. He joined Humble in 1927 following several years of geological and mining work in Joplin, Missouri. He is a past chairman of the Petroleum Division of the American Institute of Mining and Metallurgical Engineers.

Humble Conducts Lecture Series at Baytown Refinery

Humble Oil & Refining Company has inaugurated an annual lecture series, which will give selected personnel of its Baytown plant the benefit of developments in their fields. Dr. C. Price, University of Notre Dame, completed the first series in August on "High Polymer Chemistry."

The series, "Humble Lectures in Science," will be offered in two levels of scientific advancement, one at the highest level, the other to correspond to university graduate work. Dr. F. A. Matson of the University of Texas has opened the graduate series with a course in physical chemistry.

Among those who will follow Dr. Price in the 1946-47 series are Dr. E. R. Gilliland of Massachusetts Institute of Technology, whose subject is "Transference of Processes from Small to Large Scale"; Dr. R. C. Fuson of the University of Illinois, who will present "Advanced Topics in Hydrocarbon Chemistry"; and Dr. K. S. Pitzer of the University of California, who will lecture on "Spectra as Related to Structure and Thermodynamic Properties of Molecules." These lectures will be given on a full time basis for a period of one to two weeks, and personnel selected for the courses will devote full time to them.

The second or graduate-level group of lectures is designed for technical and research people who have had little or no opportunity for university graduate study. Material presented to this group is of a rather broad general nature, and will be given over a longer period of time; classes will be held once a week for periods ranging up to nine months. Lecturers at the graduate level have been invited from near-by colleges and universities, such as The Rice Institute, the University of Texas, and other Southwestern schools.

Joining Dr. Matsen about October 1 in the graduate series will be Dr. H. L. Lochte of the Univeristy of Texas, with lectures on organic chemistry.

All classes in both groups are small, and the number of technical and research personnel enrolled for each lecture is limited. Selection is upon the basis of the individual's need for the material being presented, and attendance is part of regular duties, so as to make no incursions on the employe's leisure time.

200 Attend Instrument Course at Texas A. & M.

The first annual short course on Instrumentation for the Process Industries at Texas A. & M. College attracted 200 instrument engineers and others concerned with design, operation or maintenance of process plants to College Station on September 4-6. The course placed emphasis on instrument applications to continuous fluid-flow processes. It was arranged and directed by J. D. Lindsay of the department of chemical engineering, and P. G. Murdock, professor of chemical engineering at the college. A number of companies concerned with chemical processing, and manufacturers of instruments and automatic control equipment cooperated, and provided lecturers, including the following subjects:

"The Economics of Instrumentation," C. F. Comstock, Monsanto Chemical Company; "Measurement of Liquid Flow and Level," L. K. Spink, The Foxboro Company; "Measurment of Temperature and Pressure," V. L. Parsegian, C. J. Tagliabue Division, Portable Products Corporation; "Survey of Electrochemical Measuring Devices," W. N. Greer, Leeds & Northrup Company; "General Theory of Process Control," J. G. Ziegler, Taylor Instrument Companies; "Analysis of Light-Ends Fractionation," E. D. Mattix, Cities Service Refining Corporation, Lake Charles, Louisiana; "Control of Primary Operating Variables," J. B. McMahon and R. W. Landon, Republic Flow Meters Company; "Pneumatic Circuits for Measurement and Control," W. B. Heinz, Penn Industrial Instrument Corporation; "Control Valve Characteristics," T. B. Burris, Fisher Governor Company, and "Survey of Primary Measuring Devices," D. M. Considine, Brown Instrument Company.

Presiding at various sessions were: Professor Lindsay; C. F. Tears, Process Engineers, Inc., Dallas; Porter Hart, The Dow Chemical Company, Freeport; J. A. Parker, Shell Oil Company, Houston; J. Dvoracek, Climax Engineering Corporation, Houston; M. K. Anderson, Carbide and Carbon Chemicals Corporation, Texas City, and W. H. Fortney, Humble Oil & Refining Company, Baytown.

WPRA Technical Meeting At Wichita, September 26

Western Petroleum Refiners Associations has planned its third regional technical meeting for the year to be held at Wichita, Kansas, on September 26. T. A. Helling, El Dorado Refining Company, El Dorado, Kansas, will serve as chairman of the meeting, which convenes in the Broadview Hotel at 2 p.m.

"Recent Advances in Catalytic Desulphurization of Refinery and Natural Gasolines" is to be discussed by M. W. Conn and George M. Bruno of Phillips Petroleum Company, Bartlesville, Oklahoma. Much of the data to be presented is of wartime origin and hitherto unpublished, it was said. The subject was selected by the association's manufacturing committee as one of considerable interest to many refiners in the Mid-Continent area, and especially those without catalytic-cracking facilities.

"Disposal Water Treating at Talco Refinery" is to be presented by C. G. Rook, Jr., water chemist for Talco Asphalt and Refining Division, American Liberty Oil Company, who will discuss how this problem has been successfully handled at Talco, Texas.

Discussions from the floor will follow presentation of the papers.

NGAA Returns to Dallas For 1947 Convention

The twenty-sixth annual convention of the Natural Gasoline Association of America will be held April 23-25, 1947, in the Baker Hotel, Dallas, according to William F. Lowe, secretary. Originally scheduled to be held in Tulsa preceding the International Petroleum Exposition, a return to Dallas in 1947 was decided after postponement of the exposition until 1948.

In stressing the need for early reservations, Lowe stated, "The growth in registration in recent years and the difficulty in obtaining hotel accommodations, have made it necessary to solicit the aid of all the leading hotels in meeting the requirements. Both the Baker and Adolphus hotels have already agreed to reserve large blocks of rooms and will accept reservations at any time, giving preference to requests for double occupancy."

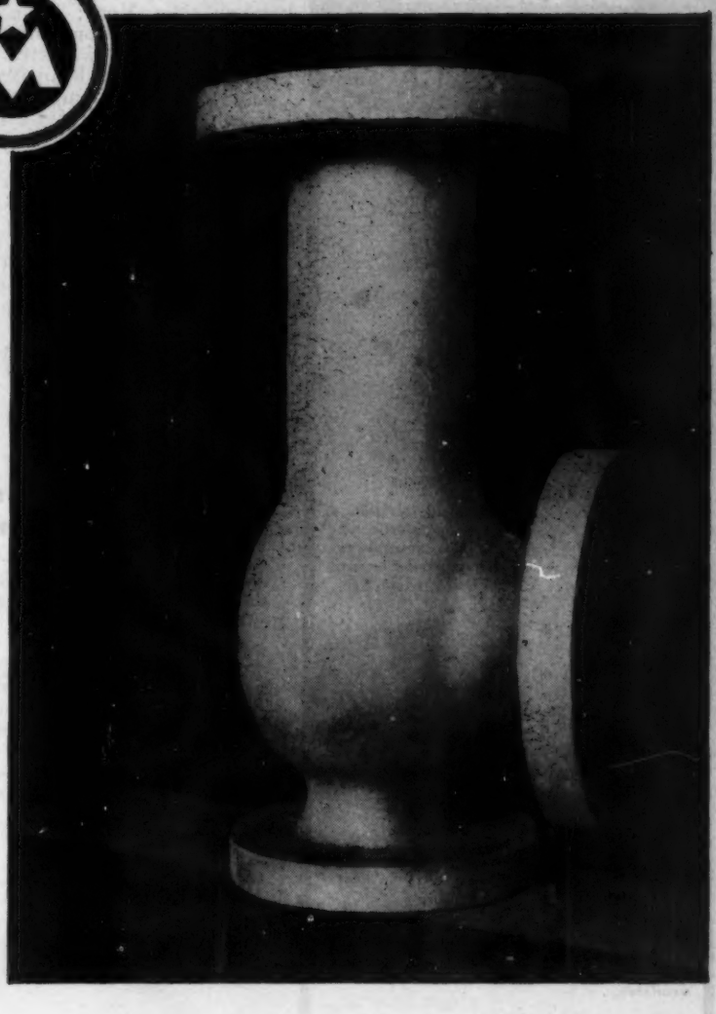
API National Oil Policy Committee Announced

Appointment of a committee on national oil policy, of the American Petroleum Institute, authorized by the board of directors at its Dallas meeting June 25, has been announced.

The committee, to be appointed annually, is to "discuss problems of national import to the industry, and from time to time submit its findings and recommendations to the board of directors."

A. Jacobsen, Amerada Petroleum Corporation, New York, is chairman. Members are: K. S. Adams, Phillips Petroleum Company, Bartlesville, Oklahoma; Robert H. Colley, The Atlantic Refining Company, Philadelphia; H. D. Collier, Standard Oil Company of California, San Francisco; Henry M. Dawes, The Pure Oil Company, Chicago; O. D. Donnell, The Ohio Oil Company, Findlay, Ohio; J. Frank Drake, Gulf Oil Corporation, Pittsburgh; W. H. Fergu-

CORROSION AND HEAT RESISTING CASTINGS



MIDVALOY 2512 (25% chromium, 12% nickel) is a cast alloy possessing high corrosion resisting qualities. It is melted in induction furnaces to exacting specification, and contains carbons to .07 max., .10 max., .16 max. or higher as needed. To meet your particular problem, other alloys such as columbium and molybdenum may be added. MIDVALOY 2512 is used extensively by the paper pulp industry for digesters, pumps, impellers, strainer fittings, valve bodies and parts.

MIDVALOY 2512 in low carbon is produced regularly in the foundry.

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PLASTIC PIPE, TUBING AND FITTINGS

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A typical installation of Saran plastic tubing that shows its extreme flexibility and fatigue strength.

Part of an installation of Saran plastic pipe that shows some of the standard fittings available.

There are few, if any, refineries, that could not save time, trouble and money by installing Saran plastic pipe and tubing for those piping systems for which it is so peculiarly well fitted to perform an efficient and lasting job. Consult our Engineering Department or write for Catalog No. PT 46.

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son, Continental Oil Company, Denver; Walter S. Hallanan, Plymouth Oil Company, Pittsburgh; B. A. Hardey, Independent Petroleum Association of America, Shreveport, Louisiana; George A. Hill, Jr., Houston Oil Company of Texas, Houston; Eugene Holman, Standard Oil Company (New Jersey), New York; W. F. Humphrey, Tide Water Associated Oil Company, San Francisco; B. Brewster Jennings, Socony-Vacuum Oil Company, New York; W. Alton Jones, Cities Service Company, New York; John M. Lovejoy, Seaboard Oil Company of Delaware, New York; A. C. Mattei, Honolulu Oil Corporation, San Francisco; J. Howard Pew, Sun Oil Company, Philadelphia; Frank M. Porter, Mid-Continent Oil & Gas Association, Oklahoma City; W. S. S. Rodgers, The Texas Co, New York; Charles F. Roeser, Roeser & Pendleton, Inc., Fort Worth; Reese H. Taylor, Union Oil Company of California, Los Angeles; H. L. Thatcher, National Council of Independent Petroleum Associations, Chattanooga, Tennessee; Albert E. Watts, Sinclair Oil Corporation, New York; H. C. Wiess, Humble Oil & Refining Company, Houston; Robert E. Wilson, Standard Oil Company (Indiana), Chicago; Ralph T. Zook, The Sloan & Zook Company, Bradford, Pennsylvania.

Grebe and Doremus Offer Refinery Consulting Service

H. A. Grebe and E. P. Doremus have established engineering services as refinery and light-hydrocarbon process consultants, with offices at 919 Fannin Street, Houston 2, Texas. The new company will specialize in economic surveys, efficiency improvement, and engineering design for the smaller refiners.

H. A. Grebe was educated at The Rice Institute, securing a B.S. degree in 1929, and on graduation was employed by The Texas Company where he served as assistant to the vice president and general manager in charge of technical service for all refineries. Crebe joined the J. S. Abercrombie Company and Harrison Oil Company in 1943, as superintendent of their Old Ocean refinery near Sweeny, Texas, taking part in the construction and operation of that plant until it was closed at the end of the war with Japan.

E. P. Doremus was educated at Southwestern Louisiana Institute, securing a B. S. in chemistry in 1935, and at Louisiana State University where he obtained a M. S. in organic chemistry in 1936. He was employed by Shell Oil Company in 1936 in its research and development department where he worked until 1943 when he, too, joined J. S. Abercrombie Company and Harrison Oil Company, as chief chemist and process engineer at Old Ocean refinery, serving there until the end of the war.

250 Attend A. and I. Short Course in Gas Technology

More than 250 engineers and gas company officials were at the first annual short course in gas technology at Texas College of Arts and Industries, Kingsville, Texas. Under the direction of Dr. Frank H. Dotterweich, head of the department of engineering, and sponsored by the Southern Gas Association, the course contained more than 28 lectures. The three sections of the course consisted of natural-gas products, gas transmission, dehydration, air conditioning, dehydration of food and agricultural products, and the latest developments in gas-turbine design.

Hanneken Retires After 44 Years in Refining

George W. Hanneken, vice president of The Standard Oil Company (Ohio) retired September 1 after 44 years of activity in the petroleum refining industry. Hanneken came to the company in 1917 to assist in planning construction of the Toledo plant. This was delayed for two years because of the first World War. When the plant was completed he remained as superintendent, where he remained until 1936 when

H. A. GREBE

E. P. DOREMUS

THE MONTH...

he became general manager of the company's manufacturing department with headquarters in Cleveland. He was elected vice president in charge of manufacturing three years later.

Hanneken went into refinery work as a pipe fitter's helper when Standard Oil Company of Kansas was enlarging its plant at Neodesha, Kansas. He remained there as a pumper, boiler fireman, water-station engineer, stillman, night foreman and finally foreman of the cracking and coking stills.

Standard of Ohio Plans Lubricating Oil Plant

The Standard Oil Company (Ohio) has announced plans of expanding $8,000,000 on construction of a 2000-barrels-per-day lubricating-oil plant at its Toledo refinery. As materials become available, it is hoped work can begin in the spring of 1947 and the plant will be ready for operation in 1948.

Humble Plans Lubricants Unit at Baytown Refinery

Humble Oil & Refining Company has let contract for design and construction of a 4600-barrel-per-day solvent dewaxing unit at Baytown, Texas, to E. B. Badger & Sons Company of Boston, Massachusetts.

The Methyl-Ethyl-Ketone process, licensed by Texaco Development Corporation, will be employed. It is anticipated that the plant will be placed in operation during 1947.

Carter Oil Company Plans Billings, Montana, Refinery

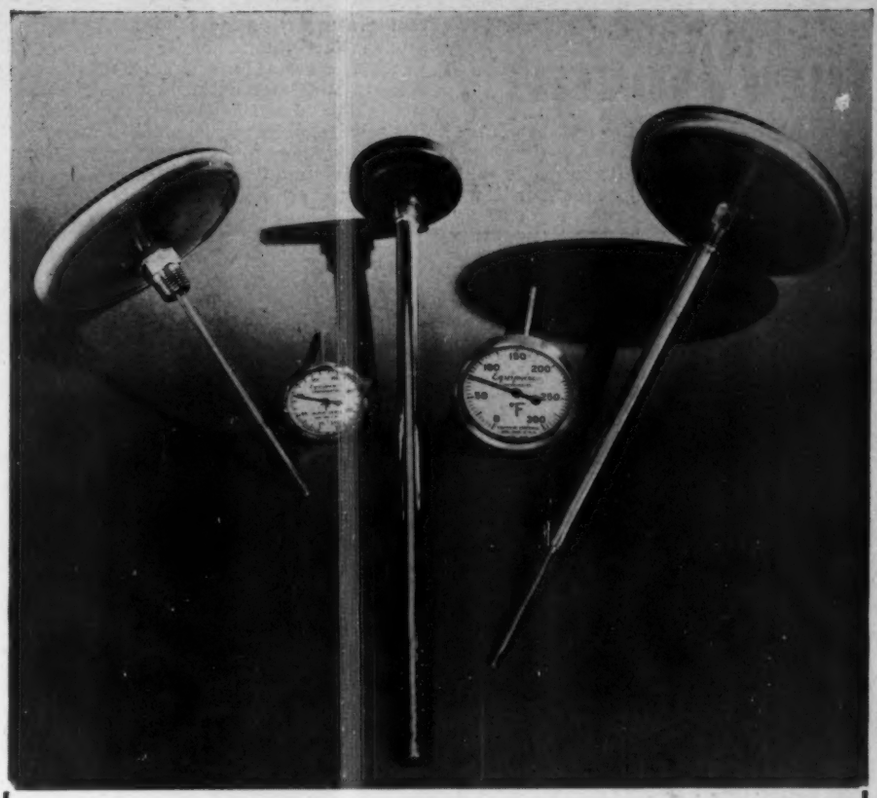
The Carter Oil Company has announced plans for construction of an 18,000-barrel refinery at Billings, Montana. It will include a catalytic-cracking unit. Plans are far advanced and construction will start as materials are available, but it probably will be 1948 before the plant will be finished, it was said.

Celanese Corporation Plans Petroleum Chemicals Lab

Construction of a $3 million research center by Celanese Corporation of America is well underway, and initial operations probably will begin within the next month, officials of the company have announced.

An outgrowth of the Celanese Chemical plant at Bishop, Texas, where chemicals are produced from natural gas, the center's prime research purpose will be for investigation and research connected with chemicals that can be derived from petroleum or petroleum derivitives. It will work toward improvement of present operation and processes used at the Bishop plant and to development of new processes for output of the same products in more efficient, less expensive ways. It also will conduct research to utilize present products in other processes to make new or improved materials. In addition, it will train technicians and operators.

Located on a 50-acre tract 6 miles west of Corpus Christi, the research



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Atlanta, Ga.—Garrard, Sudderth & Seat
Jacksonville, Florida—Richard Barthelmess Sales Co.
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Richmond, Virginia—Precision Scientific Co.
Washington, D. C.—Precision Scientific Co.
Baltimore, Md.—Precision Scientific Co.
Philadelphia, Pa.—Precision Scientific Co.

THE MONTH . . .

laboratory will have its own maintenance, construction and fine instrumentation departments and its facilities will include the latest developments in low-temperature ultimate distillation apparatus spectrometric analysis and electronic devices. There will be more than 5,000 square feet of laboratory space.

Besides the service buildings and cooling towers, present plans call for a modernistic stone main laboratory which will have unique, adjustable inside partitions to provide for enlargement of labs as needed. Pilot plants are to be provided for use in final experimentation, and provision is being made for a library and for outdoor recreation facilities. Plans are going forward under direction of Joseph E. Bludworth, director of petroleum chemicals research and development division of the company.

Premier's Golf Tournament An Annual Event

Earl Stewart of Dallas, winner of the 1946 Premier invitation tournament, putting while Wilford Wehrle, Louisville, watches.

WHEN Premier Oil Refining Company added a nine-hole golf course to its facilities at Greggton, Texas, where its first refining plant was built in 1935, the objective was to provide an opportunity for employes and their friends to enjoy recreation as they liked. But facilities for playing golf proved so attractive that now the company sponsors an invitation tournament each year. At these events personnel of the several company plants can compete with golf players especially invited.

The pictures shown here were taken at the 1946 invitation meet, which drew 128 entries, whose fees went to the Gonzales Warm Springs Foundation for Crippled Children. The year previous the entrance money went to one of the Army hospitals.

The course is laid out on 50 acres surrounding the plant. In one case the hazard is a corner of the plant yard. The course is maintained by the company gardner. Grass greens and sand traps combine to make it sufficiently difficult for expert playing.

Even the professional has his refinery connection. When "Pee Wee" Parvino is not on duty as a process unit operator in the plant he gives instructions on how to get distance and accuracy.

Sylvester Dayson (center), president of Premier Oil Refining Company, watches some of the action in the company golf tournament, whose entrance fees go to a selected charitable cause.

Public interest in the event can be estimated from attendance of 800 at the barbecue which opened the tournament August 23. The event has been carried over one of the Texas radio chains for the past two years.

New Refinery Is Planned By Egyptian Government

The Egyptian government is planning to erect a new petroleum refinery with an intake of 2800 barrels daily. The plant will be located on the same plot as the government's existing Suez refinery, which handles about 1400 barrels daily. The present refinery, although said to be nearing the end of its economic life, will be left intact, so that, if necessary, the two plants could handle 4200 barrels daily.

Experts to Germany to Study Synthetic Fuels

Four American technical experts associated with Hydrocarbon Research, Inc., are en route to Germany to investigate development in that country of synthetic liquid fuels, as representatives of the Office of Technical Services of the Department of Commerce.

The major objective of the group is to study the use by German scientists of oxygen in the synthesis of liquid hydrocarbon products, including synthetic gasoline, lubricants and the heavier hydrocarbons, as well as German methods for producing oxygen.

Members of the group are Dr. Clarence A. Johnson, Norman F. Buschow, Edward G. Engel, and Dr. Judson S. Swearingen. They are serving the OTS without compensation, and their living and traveling expenses are being paid by their company.

Refining Outlet Sought For Brazil's Production

Crude oil production in the government-owned and operated fields in Brazil averaged about 220 barrels per day during the first quarter of 1946.

Efforts to extend the fields were disappointing. Many producing wells remain shut in for lack of refining capacity.

The National Petroleum Council has authorized four Brazilian financial groups to present final proposals for construction of four refineries. Two refineries, one of 10,000 and one of 6000 barrels daily capacity, are to be built in Sao Paulo. The other two, each of 8000 barrels daily capacity, are slated for Rio de Janeiro.

It is planned to operate the refineries on imported crude, and to distribute products through the facilities of the companies now importing refined products into Brazil.

Sanders Made Technical Manager Standard of Ohio

Dr. F. J. Sanders has been appointed to the new position of technical manager of the manufacturing department of The Standard Oil Company (Ohio). He was replaced as manager of the company's No. 2 refinery at Cleveland by Forest G. Brown who has been manager of the lithograph can factory at that plant and now will combine the two positions.

Abstracts of Papers Presented Before

Division of Petroleum Chemistry, American Chemical Society

Chicago, Illinois, September 9-14, 1946

Evaluation of Laboratory Distillation Apparatus, Improved Oldshaw Glass Bubble Plate Columns, Automatic Still Heads, and Accessories.

FRANK C. COLLINS AND VERNON LANTZ, *Shell Development Company, Emeryville, California.*

Optimum Dilution in Solution Filtration.

E. J. REEVES, *Technical Department, Refining Division, Magnolia Petroleum Company, Beaumont, Texas.*

A useful relationship was derived for calculating optimum dilution in solvent dewaxing of lubricating oil stocks. This relationship is based on an empirical equation which was also developed for expressing the change in viscosity of the oil with the quantity of solvent used within the commercial ranges of operation. Observed and calculated data are shown to be in good agreement.

Polymers and Viscosity Index.

H. C. EVANS AND D. W. YOUNG.

Using data obtained during a study of the effect of temperature on the viscosity of high-molecular-weight polybutene in pure solvents the authors present a graphical interpretation of viscosity-index improvement by certain polymers and explain why the ability to improve the viscosity index is limited to polymers having definite solubility characteristics by choosing polymers of varying composition and solubility properties and blending each polymer into a number of oils varying from minus 324 to plus 105. Viscosity-index data and curves are presented showing that the relative viscosity index-improving ability of the various polymers varies with the chemical structure and the order of their potency varies with the viscosity index of the oil in which they are blended.

Evaluation of Laboratory Distillation Apparatus. Improved Oldshaw Glass Bubble-Plate Columns, Automatic Still Heads, and Accessories.

FRANK C. COLLINS AND VERNON LANTZ, *Shell Development Company, Emeryville, California.*

Novel test methods have been developed for evaluating laboratory distillation apparatus under conditions approximating actual operation rather than under ideal conditions for maximum separation. In particular, the effect of reflux ratio on efficiency and separation has been studied. Test methods for evaluating the other principal characteristics of distillation apparatus are also described in detail.

The Oldshaw glass bubble-plate column has been improved and studied using the described test methods. The column has been found to be highly satisfactory for analytical distillations of hydrocarbon mixtures because of uniformity of its operating characteristics. Automatic still heads and other accessory equipment have been developed which contribute to the uniformity of the performance of the column.

Phase Relationships in Houdry Heat-Transfer Salt Systems.

J. ALEXANDER, JR., AND S. G. HINDIN, *Houdry Process Corporation of Pennsylvania, Marcus Hook, Pennsylvania.*

A phase diagram is presented which gives in detail the initial freezing points of the system sodium-potassium-nitrite-nitrate; a portion of which represents the heat transfer material used in Houdry fixed-bed catalytic-cracking units. Other diagrams are given which show the effects on the commercially used portion of this system of certain chemical reactions which tend to take place as the mixture ages. These reactions include the oxidation of nitriteion to nitrateion, and the formation of hydroxide and carbonate ions as a result of internal oxidation-reduction followed by the absorption of water vapor and CO_2. Processes for the commercial control of these reactions are discussed. The experimental methods used in the study are also described.

The Use of Tannins and Allied Chemicals in Mercaptan Removal Treating Processes.

JOHN HAPPEL AND S. P. CAULEY, *Technical Service Division, General Laboratories, Socony-Vacuum Oil Company, Inc.*

Alkali-soluble catalysts have been found with which a rapid economical regeneration of treating solutions, used for removal of mercaptans from gasoline, can be effected through air blowing. Representative of the groups of compounds which have been investigated and found to be effective for this purpose are: Tannins, gallic acid, hydroquinone, and pyrogallol.

The influence of temperature concentrations and other variables upon oxidation rates of mercaptans is given.

Methods of using the catalysts in mercaptan-removal processes are discussed. The use of catalysts for regeneration of treating solutions presents a new approach to methods of mercaptan removal from gasolines and should result in wider application of this method of gasoline treatment.

Studies of Naphthas from Fluid-Catalyst Cracking.

C. E. STARR, JR., J. A. TILTON, AND W. G. HOCKBERGER, *Esso Laboratories, Standard Oil Company of New Jersey, Louisiana Division, Baton Rouge, Louisiana.*

The compositions of fluid catalyst cracked naphthas vary within wide limits depending upon (a) changes in operating conditions, (b) types of catalyst employed, and (c) feed stocks used. The compositions of nine naphthas are presented to illustrate the types of products obtained at low and high cracking temperatures with varying cracking severities, employing clay and synthetic catalysts with several paraffinic and naphthenic feed stocks. Composition data show that a number of valuable hydrocarbons, such as toluene, are present in the cracked naphthas to an extent making feasible their removal. The flexibility of the fluid-catalyst cracking process permits operations for production of high-quality fuels concurrent with a number of hydrocarbons that are individually valuable as raw materials for chemical manufacture.

Testing of Cracking Catalysts.

M. E. CONN AND G. C. CONNALLY.

A fixed-bed method for testing cracking catalysts is described and data are given to indicate how the test results have been applied to show the effect of catalyst quality on yields in fluid-catalyst cracking units. Other points of interest include the following: (a) cracking activity expressed as throughput for a given conversion of linear with surface area for catalysts of a given chemical composition; and (b) carbon formation is increased when the catalyst has or acquires dehydrogenating properties. The advantages and disadvantages of various types of testing units are briefly discussed.

A Mechanism of Catalytic Cracking.

R. C. HANSFORD, *Socony-Vacuum Laboratories, Paulsboro, New Jersey.*

A mechanism for the catalytic cracking of hydrocarbons in the presence of typical catalysts of the silica-alumina type is proposed, based on the acceptance of protons from the hydrocarbon by adsorbed water and on the donation of protons to the hydrocarbon by the catalyst. Water-deficient catalysts have low cracking activity, and deuterium oxide adsorbed on cracking catalysts readily exchanges deuterium for hydrogen in adsorbed hydrocarbons at temperatures far below those required for cracking. The high concentration of protons on the surface of silica-alumina as compared to that on pure silica gel further substantiates the proposed mechanism.

Molal Refractions of Mononuclear Aromatic Hydrocarbons.

NANCY CORBIN, MARY ALEXANDER, AND GUSTAV EGLOFF, *Universal Oil Products Company, Chicago, Illinois.*

The molal refractions of mononuclear aromatic hydrocarbons are additive in a homologous series, and may be expressed by an equation of the form

$$R = k + an$$

where R = molal refraction, calculated by either the Lorenz-Lorentz formula or the Gladstone-Dale formula, and n = the number of carbon atoms. The constants a and k have been evaluated by the method of least squares for 14 homologous series of mononuclear aromatic hydrocarbons.

Hydrogenation of Catalytically Cracked Naphthas for Production of Aviation Gasolines.

ALEXIS VOORHIES, JR., AND W. M. SMITH, *Esso Laboratories, Standard Oil Company of New Jersey (Louisiana Division), Baton Rouge, Louisiana, and* C. E. HEMMINGER, *Esso Laboratories, Standard Oil Development Company, Elizabeth, New Jersey.*

Hydrogenation of the olefinic constituents of catalytically cracked avia-

tion gasolines to the corresponding paraffins with hydrogenation of aromatic constituents to naphthenes results in a gasoline of improved stability and lean-mixture octane number after addition of lead, with no sacrifice in rich-mixture rating. Commercial operation of the hydrogenation process for the improvement of fluid-catalyst cracked aviation gasolines has been successfully carried out both at 150 and 3000 psi pressure using rugged sulfur-resistant catalysts of satisfactory activity and life.

The Determination of Aromatics in Petroleum Distillates.

RICHARD M. GOODING AND RALPH L. HOPKINS, *Petroleum Experiment Station, Bureau of Mines.*

Total aromatics, benzene aromatics, and naphthalene aromatics in straight-run crude-oil fractions boiling below 325° C. are determined from filtration of 100-ml. samples through silica gel. The sample is adsorbed on 175 grams of silica gel packed in a 5-foot vertical glass column and then eluted by isopropyl alcohol or some other suitable desorbent. The process separates the sample into a paraffin-naphthene mixture and an aromatic mixture. The volumes and refractive indices of the filtrate fractions indicate the amount of total aromatics in the sample. The volume and refractivity intercept (n-0.5d) determine the volume-percent of naphthalene type aromatics.

The Chemical Refining of Aromatic Hydrocarbons from Petroleum Toluene Treating.

P. L. BRANDT, R. J. LEE, AND F. T. WADSWORTH, *Pan American Refining Corporation, Texas City, Texas.*

The problems connected with the chemical refining of toluene from petroleum sources are discussed. Investigations with pure hydrocarbons have shown that diolefins are very harmful in the acid-wash color test even at very low concentrations (0.005 to 0.01 percent). Most mono-olefins have little effect on acid-wash color. Reactions with maleic anhydride have confirmed these conclusions and a toluene-treating process using 0.005 to 0.01 percent maleic anhydride has been shown to produce toluene of exceptional acid-wash color stability.

Vapor-phase treating with U.O.P. phosphoric acid catalyst is shown to be an effective method of toluene treating. Data are also presented on the variables in conventional sulfuric acid treating.

Method of Calculation of Weight Percent Ring and Number of Rings per Molecule for Aromatics.

M. R. LIPKIN AND C. C. MARTIN, *Sun Oil Company, Norwood, Pennsylvania.*

This paper continues the series of physical property analytical methods based on the temperature coefficient of density. Percent ring and average number of rings per molecule for mixtures of alkyl aromatic hydrocarbons are determined from density and density coefficient, which may be derived from mid-boiling point and density. The methods have been found accurate, on pure compound data, within 3 percent ring and 0.1 ring per molecule. Aromatics with naphthenic or olefinic side chains cause some interference but are usually not present in sufficient concentrations to affect significantly the accuracy of the methods for petroleum fractions boiling below 400° C.

Nitrogen Tetroxide Method for the Determination of Olefinic Unsaturation of Petroleum Hydrocarbons in the Gasoline Boiling Range.

G. R. BOND, JR.

Nitrogen textroxide reacts selectively with olefinic hydrocarbons, converting them into addition products of low volatility, called nitrosates. Paraffins, naphthenes, and aromatics in the gasoline boiling range are unreactive under the conditions of the test. Two methods are described for the quantitative recovery and estimation of the unreacted hydrocarbons, (a) by steam distillation or (b) by reacting the nitrosates with alcoholic KOH and K_2S to convert them into compounds soluble in 50 percent alcohol, leaving the insoluble, unreacted hydrocarbons for measurement. These methods possess several marked advantages over the usual halogen-number procedures.

The Determination of Olefins, Aromatics, Paraffins, and Naphthenes in Gasoline.

S. S. KURTZ, JR., I. W. MILLS, C. C. MARTIN, W. T. HARVEY, AND M. R. LIPKIN, *Sun Oil Company, Norwood and Marcus Hook, Pennsylvania.*

A method of analysis is described which is suitable for the determination of olefins, aromatics, paraffins, and naphthenes in gasolines. The method is more reliable than previously published methods in that it contains several improvements in analytical procedures and several internal cross checks.

The sum of the olefins plus aromatics (total unsaturates) is determined by controlled acid treatment using 30 percent P_2O_5 and 70 percent H_2SO_4. Olefins are determined by bromine number or by the N_2O_4 procedure of G. R. Bond. Aromatics are determined by specific dispersion or by subtracting the percent olefins from the total unsaturates.

Purification, Purity, and Freezing Points of 7 Heptanes, 16 Octanes, 6 Pentenes, Cyclopenten, and 7 C_9H_{12} Alkylbenzenes of the API-Standard and API-NBS Series.

ANTON J. STREIFF, EVELYN T. MURPHY, VINCENT A. SEDLAK, CHARLES B. WILLINGHAM, AND FREDERICK D. ROSSINI, *American Petroleum Institute Research Project 6, National Bureau of Standards, Washington.*

This report describes the purification and determination of freezing points and purity of the following 37 hydrocarbons of the API-Standard and API-NBS series:

PARAFFINS

2-Methylhexane	3-Ethylhexane
3-Methylhexane	2,2-Dimethylhexane
3-Ethylpentane	2,3-Dimethylhexane
2,2-Dimethylpentane	2,4-Dimethylhexane
2,3-Dimethylpentane	2,5-Dimethylhexane
2,4-Dimethylpentane	3,3-Dimethylhexane
3,3-Dimethylpentane	3,4-Dimethylhexane
n-Octane	2-Methyl-3-ethylpentane
2-Methylheptane	3-Methyl-3-ethylpentane
3-Methylheptane	2,2,3-Trimethylpentane
4-Methylheptane	2,3,3-Trimethylpentane
	2,3,4-Trimethylpentane

ALKYLBENZENES

n-Propylbenzene	1,2,3-Trimethylbenzene
1-Methyl-2-ethylbenzene	1,2,4-Trimethylbenzene
1-Methyl-3-ethylbenzene	1,2,5-Trimethylbenzene
1-Methyl-4-ethylbenzene	

MONOOLEFINS

1-Pentene	3-Methyl-1-butene
cis-2-Pentene	2-Methyl-2-butene
trans-2-Pentene	Cyclopentene
2-Methyl-1-butene	

Purification, Purity, and Freezing Points of 8 Nonanes, 11 Alkylcyclopentanes, 6 Alkylcyclohexanes, and 4 Butylbenzenes of the API-Standard and API-NBS Series.

ANTON J. STREIFF, EVELYN T. MURPHY, JANICE C. CAHILL, HELEN F. FLANAGAN, VINCENT A. SEDLAK, CHARLES B. WILLINGHAM, AND FREDERICK D. ROSSINI, *American Petroleum Institute Research Project 6, National Bureau of Standards, Washington.*

This report describes the purification and determination of freezing points and purity of the following 29 hydrocarbons of the API-Standard and API-NBS series:

PARAFFINS

n-Nonane
2,2,5-Trimethylhexane
2,4,4-Trimethylhexane
2,2,3,3-Tetramethylpentane
2,2,3,4-Tetramethylpentane
2,2,4,4-Tetramethylpentane
2,3,3,4-Tetramethylpentane
3,3-Diethylpentane

ALKYLCYCLOPENTANES

Ethylcyclopentane
1,1-Dimethylcyclopentane
cis-1,2-Dimethylcyclopentane
trans-1,2-Dimethylcyclopentane
trans-1,3-Dimethylcyclopentane
n-Propylcyclopentane
Isopropylcyclopentane
1,1,2-Trimethylcyclopentane
1,1,3-Trimethylcyclopentane
cis,cis,trans-1,2,4-Trimethylcyclopentane
cis,trans,cis-1,2,4-Trimethylcyclopentane

ALKYLCYCLOHEXANES

Ethylcyclohexane
1,1-Dimethylcyclohexane
cis-1,2-Dimethylcyclohexane
trans-1,2-Dimethylcyclohexane
cis-1,4-Dimethycyclohexane
trans-1,4-Dimethylcyclohexane

ALKYLBENZENES

n-Butylbenzene
Isobutylbenzene
sec-Butylbenzene
tert-Butylbenzene

Purification and Sealing "In Vacuum" of NBS Standard Samples of Hydrocarbons.

BEVERIDGE J. MAIR, DOMENIC J. TERMINI, CHARLES B. WILLINGHAM, AND FREDERICK D. ROSSINI, *Hydrocarbon Standard Samples Laboratory, National Bureau of Standards, Washington.*

This report describes the procedure and apparatus used in the purification and selling "in vacuum" of the following 19 NBS standards samples of hydrocarbons:

n-Pentane	Cyclohexane
Isopentane	Methylcyclohexane
n-Hexane	Benzene
2-Methylpentane	Toluene
3-Methylpentane	Ethylbenzene
2,2-Dimethylbutane	o-Xylene
2,3-Dimethylbutane	m-Xylene
n-Heptane	p-Xylene
2,2,4-Trimethylpentane	Isopropylbenzene
Methylcyclopentane	

The Use of the Grignard Reaction in the Synthesis of Hydrocarbons.

J. DONALD GIBSON, FRANK W. HAECKL, JOHN C. BELTZ, LOUIS C. GIBBONS, GRANT CRANE, ROBERT H. ARROWSMITH, HARRY E. RISHER, GERALD T. LEATHERMAN, HENRY H. CHANAN, ROBERT W. SHORTRIDGE, AMOS TURK, JULES I. SHAPIRO, THOMAS H. NEWBY, EDGAR A. CADWALLADER, MELVERN C. HOFF, WALTER C. EDMISTON, THOMAS S. HODGSON, KENNETH W. GREENLEE, ALBERT L. HENNE, AND CECIL E. BOORD, *Department of Chemistry, The Ohio State University, Columbus, Ohio.*

In the course of an extended research on the components of gasoline, sponsored by the American Petroleum Institute and the Research Foundation of this university, 153 hydrocarbons have been synthesized and/or purified in quantities of half a pint to 5 gallons. Of this total, 61 were prepared by schemes involving the Grignard reaction in one or more steps and some of them were prepared by 2 or 3 such

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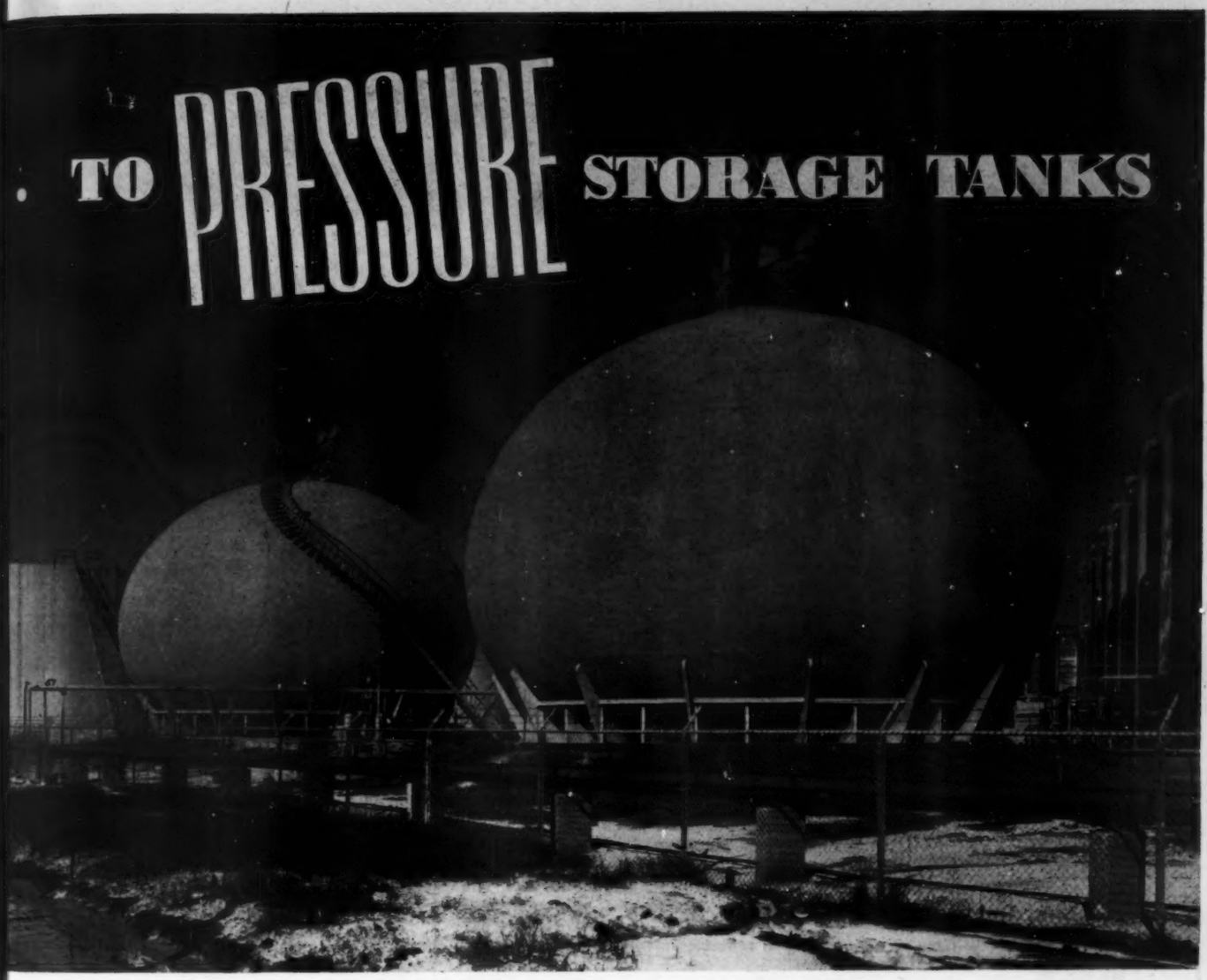

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Crude oils produced in Gulf Coast fields are processed at this refinery. When the first crude was charged in 1931, the capacity was 5,000 bbls. per day. Now the rated capacity is approximately 30,000 bbls. daily.

Present facilities at this refinery include crude topping, thermal cracking, catalytic cracking, thermal reforming, light hydrocarbon recovery and petroleum solvent units.

The catalytic cracking unit, which is served by the two catalyst bins, is used in the production of premium motor fuel. It has a rated capacity of 11,-500 bbls. of gas-oil charging stock per day but has been operated at a charging rate of 14,000 bbls.

Hortonspheroids, just one of several types of pressure storage tanks which we build, are recommended for storing products that range in volatility from motor gasoline to natural gasoline.

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San Francisco 11.....1251-22 Battery St. Bldg.
Tulsa 3.....1626 Hunt Bldg.
Cleveland 15.....2234 Guildhall Bldg.
New York 6.....3302-165 Broadway Bldg.
Philadelphia 3.....1608-1700 Walnut St. Bldg.
Washington 4.....703 Atlantic Bldg.
Detroit 26.....1520 Lafayette Bldg.

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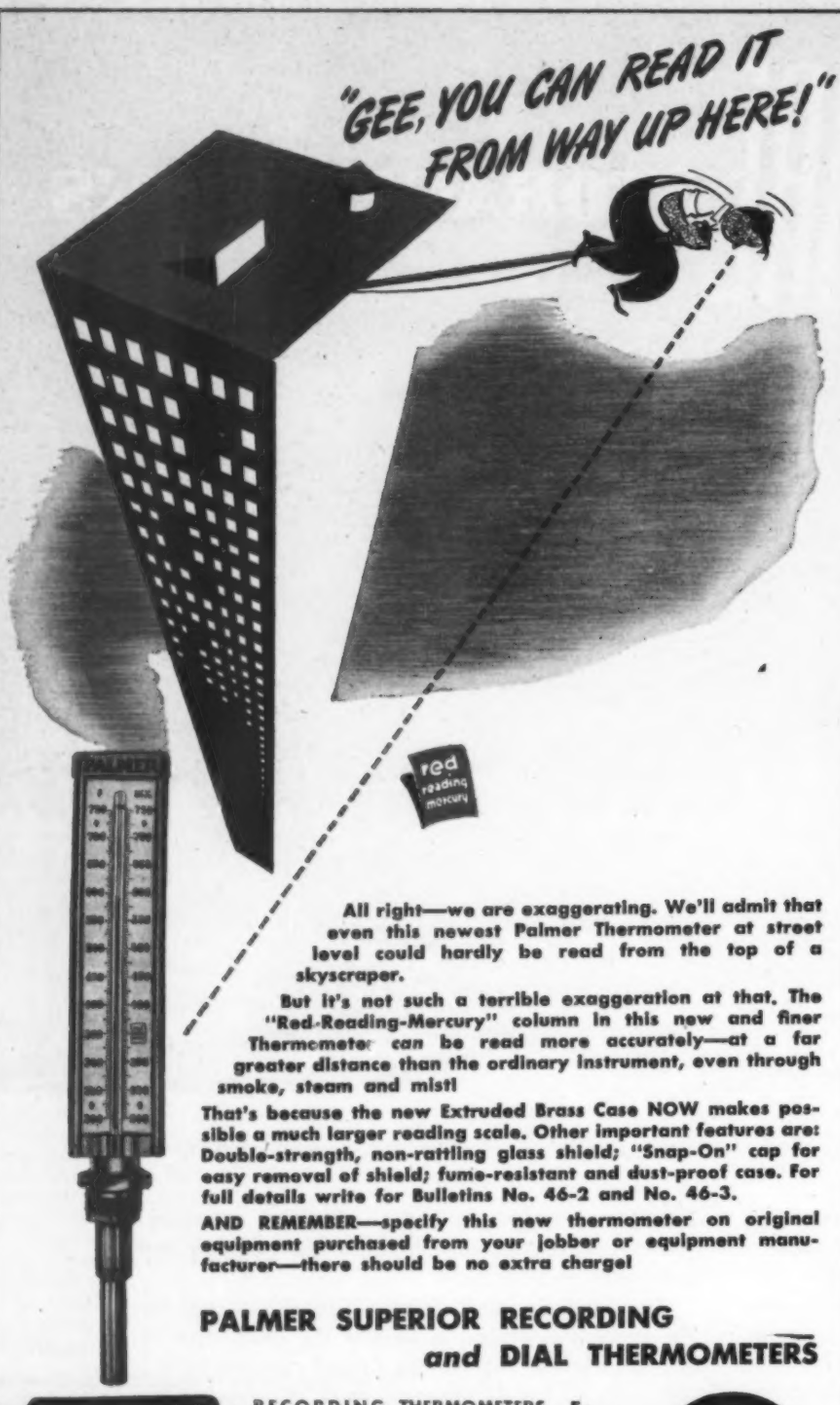

All right—we are exaggerating. We'll admit that even this newest Palmer Thermometer at street level could hardly be read from the top of a skyscraper.

But it's not such a terrible exaggeration at that. The "Red-Reading-Mercury" column in this new and finer Thermometer *can* be read more accurately—at a far greater distance than the ordinary instrument, even through smoke, steam and mist!

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methods. Eight years of experience with several distinct types of Grignard reactions have brought out the following points:

1. Metal reactors are highly efficient and satisfactory. Copper-lines vessels are best, the yields being equal to or better than those obtained from the same reactions in glass. The yields obtained in steel reactors are a little lower, but are acceptable.

2. Alkyl chlorides can be used as well as bromides after the techniques for starting the reaction with magnesium have been mastered. Powerful stirring and careful attention to dilution are needed, but in some respects the chlorides are actually superior.

3. Synthetic ether can be bought (Carbide and Carbon) which is pure enough to be used directly for the Grignard reaction medium.

4. Purity of the products depends both on the quality of the reagents and on the use of proper techniques in working up the crudes.

5. When the techniques employed are chosen with respect to their special reactivity, allylic halides are highly versatile and useful in the Grignard reaction.

Experimental Equilibrium Constants for the Isomer Hexanes.

B. L. Evering and E. L. d'Ouville, *Research Laboratories of Standard Oil Company (Indiana).*

The experimental equilibria for the isomeric hexanes were determined in the range 21° to 204° C. using activated aluminum halide catalysts on a 67° C.-end-point light naphtha stock containing both pentanes and hexanes. The present work agrees with the values calculated from the heat content by Rossini, Prosen, and Pitzer for n-hexane and 2,3-dimethylbutane but there is considerable discrepancy for 2,2-dimethylbutane and the methylpentanes. However, there is substantial agreement in the ratio of 2-methylpentane to3-methylpentane.

As the light naphtha contained pentanes the equilibria for these were simultaneously determined. The temperature coefficient for the pentane equilibrium was found to be somewhat greater from this work than that found by Pines and coworkers. This is due to lower values at 161° and 204° C. Hydrogen and extensive side reactions were not found to have an appreciable effect on the equilibria.

Harger Made Manager At El Segundo Refinery

D. K. Harger has been appointed manager of all refining divisions at the El Segundo refinery of Standard Oil Company of California.

Harger joined the company in 1926 and has spent 13 of his 20 years connections at El Segundo, and recently was superintendent of the light oils division at Richmond refinery.

Easton Joins Great National Oil Corporation

Harry D. Easton, Jr., has been elected vice president of Great National Oil Corporation, with headquarters in Shreveport. Among other activities the company has a natural gasoline plant at Ida, Louisiana.

Easton spent five years in military service and rose to the rank of colonel.

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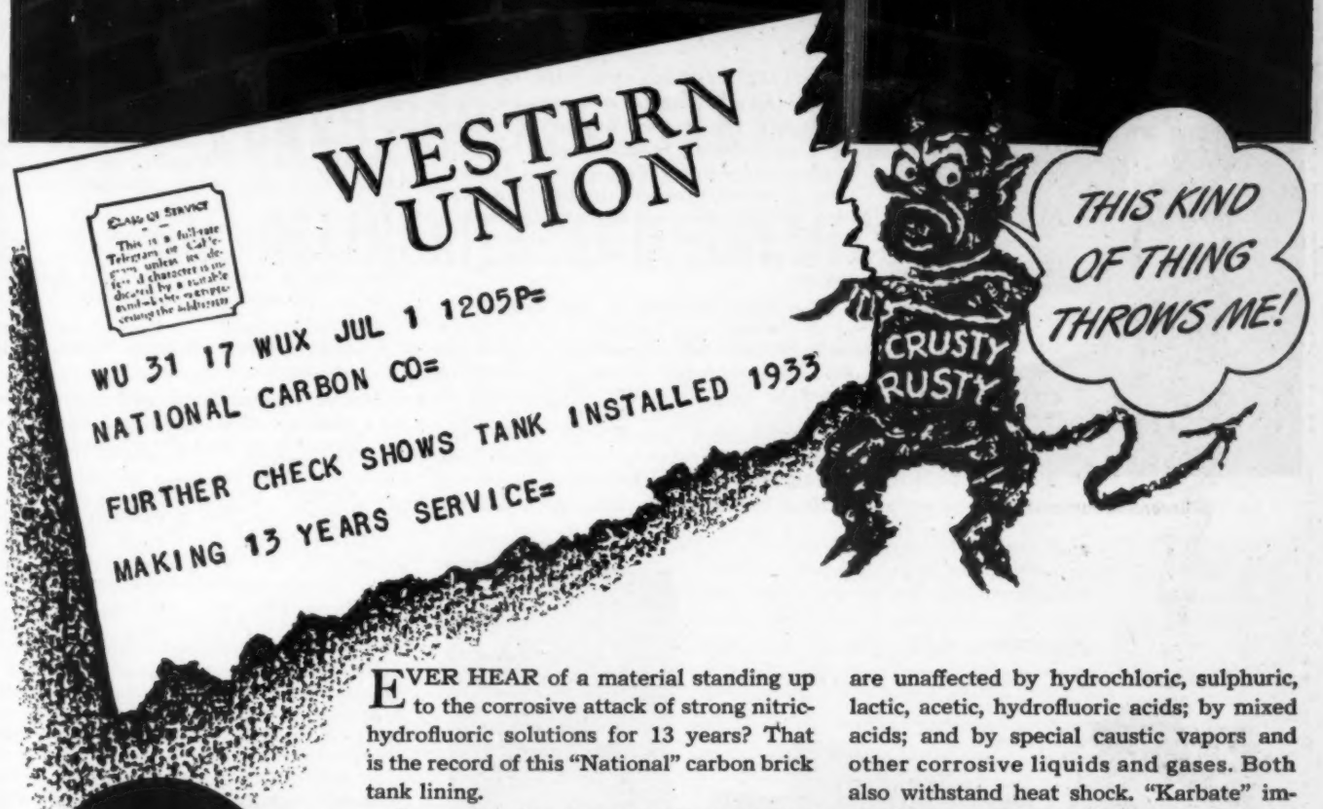
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New building housing the Physics Division in the Riverside laboratories.

UOP Formally Opens Physics Laboratory at Its Riverside Plant

Recording micrometer.

IN THE research activities of Universal Oil Products Company physical chemistry has grown up and now has its own building, which was formally opened August 1. This physics laboratory is the thirteenth building on the plot of 27 acres at Riverside, Illinois, where 260 workers are engaged in major phases of petroleum refining research.

The new building is the outgrowth of a development which had its start in 1939 with the adaptation of catalysis as a means of petroleum conversion. In order to determine the effectiveness of catalysts it was desirable to examine the substances physically, hence the powerful microscopes and other aids for the physics worker.

More recently devices such as the mass spectometer have been so adapted to examination of hydrocarbons that physics now plays a role in control over processes. Consequently the control laboratory is drawing on what has been found only in the research laboratory.

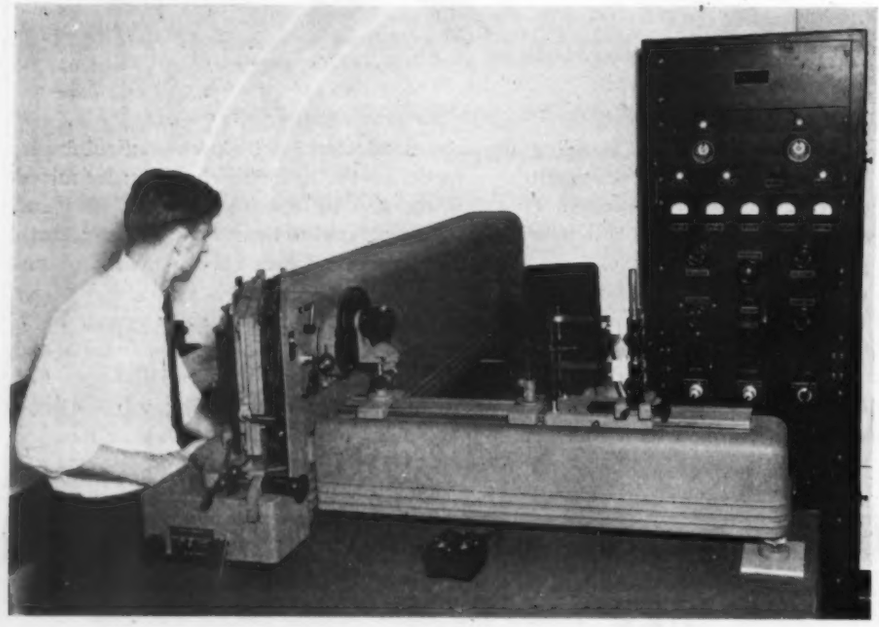
The scope of physics in Universal Oil Products Company activities was described in the comprehensive description of the new laboratory as:

1. Improvement of known catalysts and a study of their fundamental properties.
2. The identification and quantitative analysis of gaseous and liquid hydrocarbons and the products made from them.
3. General physio-chemical studies of interest to the petroleum industry.

Universal Oil Products Company, owned by the American Chemical Society, is devoted to engineering and research within the petroleum industry and its activities are primarily in behalf of the smaller refining companies which do not have research facilities. D. W. Harris, president, spoke briefly of this function in his greeting to representatives of the trade press and newspaper representatives who visited the Riverside laboratory August 1 to view the new facilities as well as to see the other equipment.

"UOP," he pointed out, "established itself as the research and engineering headquarters for the smaller refining companies. Although its facilities are available to the whole refining industry, it will continue to stress its service and relationship to the smaller organizations."

Following inspection of the laboratory and lunch, guests of the company were entertained at the Union League Club in Chicago. In addition to the equipment shown in the accompanying pictures the new physics building houses an ultra violet spectrophotometer, a Raman spectroscopy unit; a



Quartz emmission spectrograph.

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Recent innovations in operating conditions in refineries, petro-chemical plants, and plastic manufacture have led to countless new products with accompanying severe problems of corrosion and product contamination. Bridgeport's modern copper-base alloys are the answer to many of these problems. However, in cases where a single metal or alloy is unable to cope with the situation, Duplex Tubing, two tubes of entirely different materials drawn together, simplify many a complex corrosion condition.

In oil refineries arsenical Admiralty continues to be most popular. Where sulfur corrosion is especially troublesome, arsenical Muntz*, with its high zinc content, is standing up nicely against both the oil and the circulating water. For exceptionally severe corrosion from the circulating water side, either salt or polluted fresh water, Cuzinal (aluminum brass) Duronze IV** (aluminum bronze) and Cupro Nickel are available. In salt refineries Duronze IV pipes are successfully used for conveying hot brine solutions.

The ever-widening use of industrial refrigeration has focused attention on the possibilities for extending the life of refrigeration tubing. Here Duplex Tubing has been outstandingly successful in ammonia refrigeration systems and in condensers and heat exchangers handling ammonia compounds. In such applications Duplex Tubing with steel to the ammonia side and copper or a copper-base alloy to the water side is most popular.

Duplex Tubing involving such components as copper-base alloys in combination with aluminum, low carbon steel, monel, nickel or stainless are finding greater use in the chemical, food and pharmaceutical fields. Duplex Tubing is also preventing product contamination and is showing promising results when used in processes involving elevated temperatures.

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cited the development of a metallic aluminum catalyst for the catalytic dehydrofluorination of alkyl fluorides. Such a catalyst was desired for in the hydrogen fluoride alkylation process a small amount of hydrogen fluoride remains in the finished alkylate in the form of alkyl fluorides. In the prosecution of this project a small laboratory test unit was constructed in which alkylate containing combined hydrogen fluoride could be passed over solid contact materials, to determine their efficiency in splitting off the combined HF. The catalyst finally developed consisted of 1-inch raschig rings made from metallic aluminum strips. The rings were made active for dehydrofluorinating alkyl fluorides by etching them in a solution of hydrochloric acid, washing and drying. These rings have been used in large quantities in several alkylation plants and have resulted in the recovery of hydrogen fluoride valued at many thousands of dollars."

mass spectrometer; an X-ray diffraction machine and an electron microscope. All equipment was explained by the men who operate it.

Another quotation from the description of the laboratory gives an outline of function of physical examination in this description of the general laboratory part of the physics laboratory:

"The equipment in this laboratory includes an Andrews' apparatus for studying P-V-T relations of hydrocarbons and their critical properties, a surface-area unit which is used for determining the surface properties and pore size distributions of catalysts, several optical microscopes, a fairly extensive supply of chemical glassware used in preparing catalysts on a small scale, and a group of muffle furnaces for calcining catalysts at temperatures up to 2500° F. in various atmospheres.

"As typical of the work done on catalysts in this laboratory may be

infrared absorption spectrometer.

General laboratory, Universal Oil Products Company, at Riverside, Illinois.

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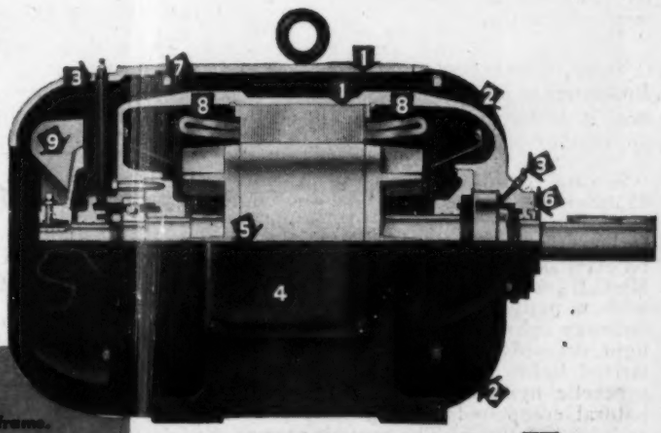

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Photostat copies of original articles will be supplied at cost by The Leslie Laboratories. Complete or limited bibliographies covering special topics by title, by abstracts, or in complete manuscript, will also be prepared and furnished at reasonable cost by the Laboratories.

Fundamental Physical and Chemical Data

The Vapor Pressures and Saturated Liquid and Vapor Densities of the Isomeric Hexanes. W. B. KAY. *Jour. Amer. Chem. Soc.* **68** (1946) pp. 1336-39.

The vapor pressures and saturated liquid and vapor densities of the five isomeric hexanes were determined over a temperature range from their atmospheric boiling points to their critical points. The static method of measuring vapor pressure was employed. The preparation and purification of the substances studied is described. Equations are given representing the vapor-pressure data and also the density data. Values of the constants in these equations are given in tabular form. Critical temperatures and pressures of the hydrocarbons are tabulated. These were determined by the disappearance-of-the-meniscus method.

Some Observations on the Optical Properties of Long-Chain Normal Paraffins. B. HUBBARD. *Am. Mineral* **30** (1945) pp. 645-72.

Normal paraffins from C_{21} to C_{33} from 95 different sources, some very pure and others mixtures of two or three hydrocarbons, were crystallized at 20-25° C. on glass slides from CCl_4, C_6H_6, MeC_6H_5, $Me_2C_6H_4$, and the slides were examined with a petrographic microscope using ordinary light as well as plane-polarized light, crossed nicols, and convergent polarized light. The crystals of the pure synthetic hydrocarbons, as well as the natural compounds are optically active and dextrorotatory. The crystals are normally orthorhombic, and approach hexagonal symmetry. No monoclinic crystals were found. The optical activity is best explained by assuming a spiral molecular chain rather than the more generally accepted idea of a zig-zag chain.

Composition of Vapors from Boiling Binary Solutions. D. F. OTHMER AND F. ROBERT MORLEY. *Ind. & Eng. Chem.* **38** (1946) pp. 751-57.

The increased use of distillation under pressure makes it desirable to have available a method and apparatus for the determination of vapor-liquid equilibrium data at pressures above atmospheric. The authors constructed an apparatus for use at pressures up to several hundred pounds per square inch. This is described in some detail, and the operating procedure is outlined. Data on the binary systems methanol-water and acetone-water are reported at pressures up to 200 psia. Correlations of these data with each other and with data from the literature at atmospheric pressure were found to be good. Acetone-water has a constant boiling mixture at higher pressures, as was predicted from the shape of the atmospheric X, Y curve, since this curve is concave upward. The data secured in the course of the work are presented in considerable detail in both tabular and graphical form.

Calculation of Specific Dispersion of Pure Hydrocarbons and Petroleum Fractions. M. R. LIPKIN AND C. C. MARTIN. *Ind. & Eng. Chem.*, Anal. Ed. **18** (1946) pp. 433-36.

The authors present an equation for the calculation of specific dispersion of pure hydrocarbons and petroleum fractions from common physical properties, such as density, refractive index, and approximate molecular weight, which properties can be estimated from mid-boiling point and density or viscosities at 100° and 210° F. It is thus possible to calculate the specific dispersion of a highly colored sample and to obtain dispersion values in a laboratory in which no dispersion instrument for direct measurement is available. The accuracy of the specific dispersion calculated by the method presented is comparable with the accuracy of measurements obtained by use of the Abbe instrument, i.e. ± 3 units. Detailed data are presented in five tables, and a bibliography of 18 references is included.

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The National Registry of Rare Chemicals, Armour Research Foundation, 33rd, Federal, and Dearborn Sts., Chicago 16, Illinois, has received urgent requests for the chemicals listed below. If anyone has one or more, even if only one gram quantities, please inform the Registry.

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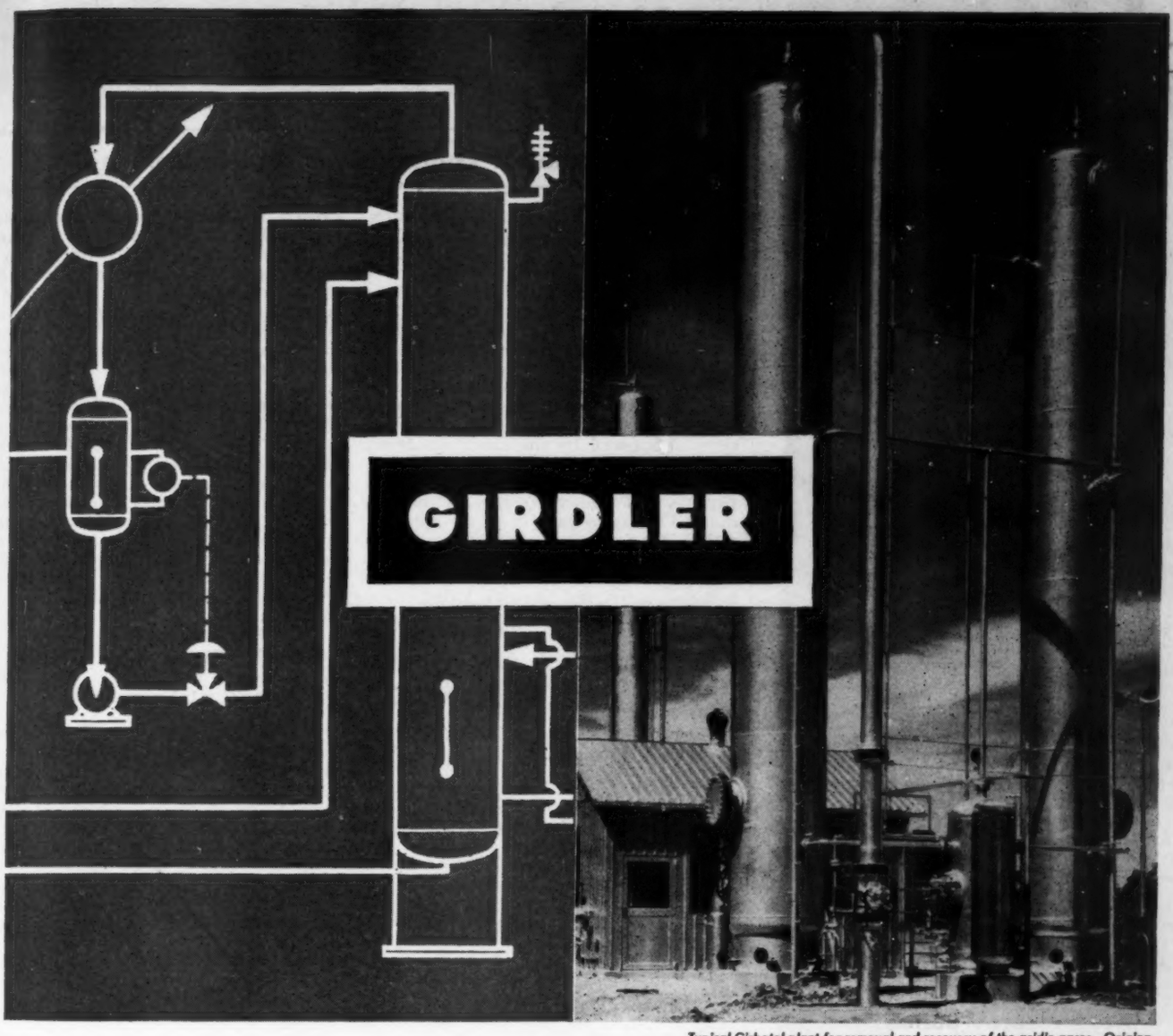
1,2-Pentanediol
1,3-Pentanediol
1,5-Pentanediol
2,3-Pentanediol
Neovaleraldehyde
Methyl ethyl ether
Hydrindene (1 kg.)
4-Nitro pyrocatechol
Alpha and gamma pyrone
3,5-Dihydroxy toluene
3,5-Dinitrotoluene
Propyl nitrate
Carnaubyl alcohol
Ceryl alcohol
1,2,4,5,-Tetrahydroxy benzene

The Molecular Refractions of the Higher Acetylenic Hydrocarbons. G. F. HENNION AND T. F. BANIGAN, JR. *Jour. Amer. Chem. Soc.* **68** (1946) pp. 1381-82.

The authors note that the calculated molecular refractivities of organic compounds usually do not agree exactly with the observed values given by the Lorenz-Lorentz equation. This is particularly true among the higher acetylenes. Methods of calculation that have been used previously are discussed. In order to estimate the expected refractions more closely, the authors used an entirely different method of calculation based on summing the average contributions of the various types of atoms in the molecule. Several examples are given showing the contributions of different types of atoms, and the physical constants and molecular refractions of the higher acetylenes are tabulated, including density, refractive index, observed molecular refraction, and molecular refraction calculated by the old method and by the new method presented by the authors.

Metastable Ions Formed by Electron Impact in Hydrocarbon Gases. J. A. WHIPPLE, R. E. FOX AND E. V. CONDON. *Phys. Rev.* **69** (1946) pp. 347-56.

The non-integral masses appearing in the mass spectra of various hydrocarbons can be explained by the spontaneous dissociation of some of the ions into fragments of lighter mass after they have been accelerated and emerge from the ion gun. By using an energy filter, an energy analysis of the non-integral masses in n-butane, butadiene, and ethane were made. The values obtained are in agreement with those predicted on the basis that they are caused by metastable ions. Variations of the pres-



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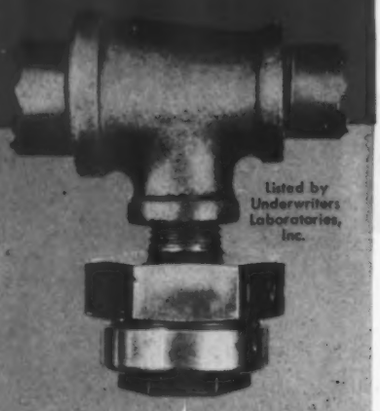

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sure and electrode potentials confirms that the dissociation is spontaneous. Metastable ions are apparently formed frequently in the ionization and dissociation of hydrocarbons, and is shown by the authors to occur in the case of ethane, propane, 1,3-butadiene, butene-1, cis-butene-2, isobutylene, normal butane, isobutane, pentene 2, normal pentane, isopentane, and methylcyclopentane.

Chemical Compositions and Reactions

Di-t-butylacetylene. G. F. Hennion and T. F. Banigan, Jr. *Jour. Amer. Chem. Soc.* **68** (1946) pp. 1202-4.

Di-t-butylacetylene, previously unknown, was prepared in order to determine its physical properties and to study the behavior of the hindered triple bond in typical addition reactions. The hydrocarbon was found to have physical properties consistent with its compact, symmetrical structure, and chemical properties influenced by the steric screening of the triple bond. The hydrocarbon was hydrogenated to sym-di-t-butylethylene and thence to sym-di-t-butylethane. Oxidation gave di-t-butyldiketone rather than pivalic acid. Attempts to hydrate the triple bond catalytically were unsuccessful. Reduction by sodium in liquid ammonia likewise failed. Bromine added slowly to give a dibromo derivative.

Allylic Chlorides. II. Catalytic Hydrolysis and Characterization of 1,3-Dichloropropene. L. F. Hatch and G. B. Roberts. *Jour. Amer. Chem. Soc.* **68** (1946) pp. 1196-98.

Although 1,3-dichloropropene has become readily available through the development of the high temperature substitutive chlorination of propylene, the two geometrical isomers (cis and trans) have not as yet been characterized as to their geometrical configuration. The present study was made to effect this characterization. 1,3-Dichloropropene was hydrolyzed using a hydrochloric acid solution of cuprous chloride. The low-boiling, or alpha, isomer hydrolyzed more slowly than the high-boiling, or beta, isomer. From this it was concluded that the effect of the cuprous chloride-chloride ion complex is through the double bond, that the action of the complex is the rate-controlling step, and that the configuration of the isomer directly affects the rate of reaction. The trans structure was assigned to the alpha isomer and the cis structure to the beta isomer.

Autoxidation of Furfural. A. P. Dunlop, P. R. Stout and S. Swadesh. *Ind. & Eng. Chem.* **38** (1946) pp. 705-8.

Color and acid formation in furfural at room temperature were shown to be caused by autoxidation. In normal storage, furfural slowly darkens, a change which is accompanied by formation of small amounts of acid which have sometimes been thought to be acetic acid or furoic acid. In many respects furfural reacts analogously to benzaldehyde. However, in its oxidation the rate of oxidation is slower and is interrupted at a lower level of acidity than in the case of benzaldehyde. A mixture of acids is formed from furfural, not merely acetic acid or furoic acid, as has been previously thought. Changes can be prevented by storing furfural in an oxygen-free atmosphere, or can be effectively inhibited by adding to the furfural a small quantity of any one of a variety of basic substances such as tertiary amines, alkali metal soaps or phenolic antioxidants. Water causes a partial inhibition of the autoxidation.

Dealkylation of Dialkylbenzenes. W. M. Kutz and B. B. Corson. *Ind. & Eng. Chem.* **38** (1946) pp. 761-64.

By conducting the dealkylation of a polyalkylbenzene in the presence of sufficient benzene it is theoretically possible to obtain as many moles of monoalkylbenzene as there are alkyl groups in the original polyalkylbenzene. Diethylbenzene was dealkylated under pressure over an alumina-silica catalyst in the presence of a large excess of benzene. Operating at 400° C., 500 psi, and 2 liquid hourly space velocity on a mixture of 1 mole of diethylbenzene and 10 moles of benzene, the yield per pass was 1 mole of monoethylbenzene, and the ultimate yield was 1.8 moles of monoethylbenzene as compared to a theoretical ultimate yield of 2. Compared with diethylbenzene, xylene was more difficult to dealkylate. Diisopropylbenzene plus 10 moles of benzene gave 1.6 moles of monoisopropylbenzene per pass. The effect of the operating variables of time, temperature, pressure, and amount of benzene was studied. Operation under pressure was found to be preferable to operation at ordinary pressure as regards conversion per pass, ultimate yield, and catalyst life. The apparatus used in the work is illustrated and described, and the data secured are summarized in tabular form. A bibliography of 12 references is included.

Manufacture: Processes and Plant

Desalting of Petroleum by Use of Fiberglas Packing. T. A. Burtis and C. G. Kirkbride. *Trans. Amer. Inst. Chem. Engrs.* **42** (1946) pp. 413-439.

The quantity of salt in crude petroleum as delivered to refiners has increased sharply, and today, a salt content, expressed as sodium chloride, up to 200 pounds per 1000 barrels of crude oil and over is common. The increase in salt concentration has been accompanied by rapid corrosion of refining equipment and has had bad effects on the refining processes themselves. Salt is a concentrated aqueous solution emulsified in the oil. A study was made of the feasibility of using beds of fine glass fibers for desalting petroleum. The oil is washed with water at a temperature of 250° F. and oil and water are passed through beds of glass fibers averaging 0.00028 inches in diameter to coalesce the emulsified water. The desalted oil is

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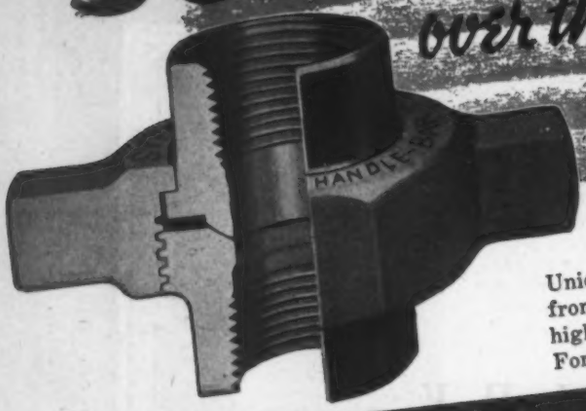

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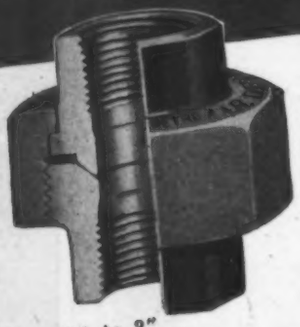

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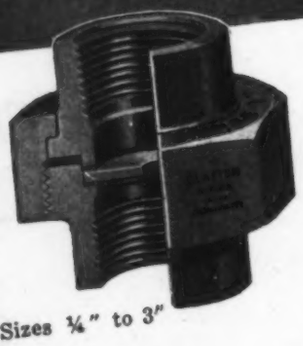

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separated from the aqueous phase. The water phase is recycled in order to conserve heat. Only enough fresh water is used to maintain the salt content of the recycled water at a maximum of 3 percent of salt by weight. It has been found that the salt content can be reduced to less than 5 pounds per 100 barrels by proper control of temperature, superficial velocity, density of the Fiberglas beds and total depth of the beds. In general, temperature should be at least 275° F. Maximum permissible superficial velocities ranged from 0.25 to 1.0 feet per minute depending upon the type of oil handled. The effectiveness of the packing increased rapidly with increasing density of the beds up to about 15 pounds per cubic foot. A total depth of the beds of fibers of 5 inches is adequate for desalting to less than 5 pounds per 1000 barrels. About 90 percent of the salt is removed in the first two inches of the bed. The authors believe the process is effective and attractive from an economic standpoint for commercial application.

Purification of Butadiene with Cuprous Salt Solutions. C. E. Morrell, W. J. Paltz, J. W. Packie, W. C. Asbury and C. L. Brown. *Trans. Amer. Inst. Chem. Engrs.* **42** (1946) pp. 473-94.

The purification of butadiene by means of cuprous salt solutions has resulted in a commercial process that played a major part in supplying butadiene for wartime synthetic rubber production. At present plants in the United States and Canada have a rated annual capacity of approximately 260,000 tons of butadiene. This represents about 55 percent of the total capacity of the plants built by the government for the production of butadiene from petroleum sources. The impurities present in crude butadiene from petroleum sources are listed in tabular form. Preparation of the ammoniacal cuprous acetate solutions used is described. The solubilities of mono- and diolefins in ammoniacal cuprous acetate solutions at 32° F. and 0.5 atmosphere hydrocarbon partial pressure are tabulated. The problem of removing the acetylene from the butadiene material is discussed. The heats of absorption of the gaseous unsaturated hydrocarbons in ammoniacal cuprous acetate solutions are tabulated. All processes include an absorption step in which the butadiene, together with small quantities of other unsaturated hydrocarbons, and especially the butenes, are dissolved in the solvent. Second, a butadiene enrichment step in which the unsaturated hydrocarbons other than butadiene are stripped from the solvent, and lastly a desorption step in which butadiene of high purity is recovered from the solvent. Factors of importance in connection with each of these steps are discussed. Commercial plants are listed as to operating company, location, and capacity.

Octane Rating Improvement of Olefinic Gasolines by Isomerization. L. Berg, G. L. Sumner, Jr. and C. W. Montgomery. *Ind. & Eng. Chem.* **38** (1946) pp. 734-37.

The olefins in an olefinic gasoline can be isomerized with a minimum of side reactions such as cracking, hydroforming, treating, aromatization, and cyclization. The best results are obtained at 375-425° C. reaction temperature and 0.3-1.3 volume per volume of catalyst

HEAD START in the field

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The twelve "orange-peel" segments of the hemispherical head could have been piled on freight cars and welded together at the refinery. But instead, the segments were completely assembled in the B&W plant to assure accurate fit. All but two final welds on the head were completed in the B&W shop, where facilities for doing this work were more favorable than in the field.

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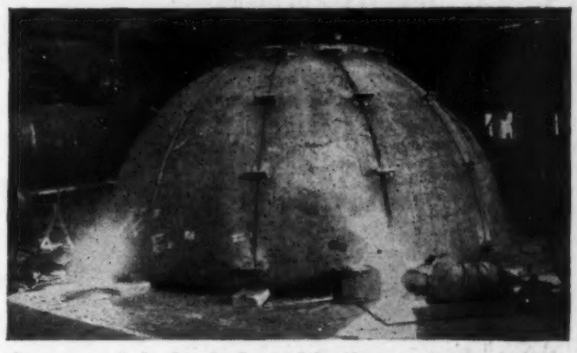
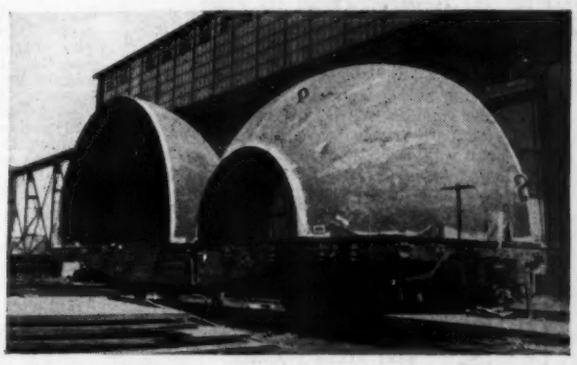
Shell plates for top section of tower were shipped in parts as shown above, for field erection.



Segments of the hemispherical head were drawn together and shop-welded into two sections at the B&W plant.



Only four welds were required to assemble the two sections of the tower head in the field.

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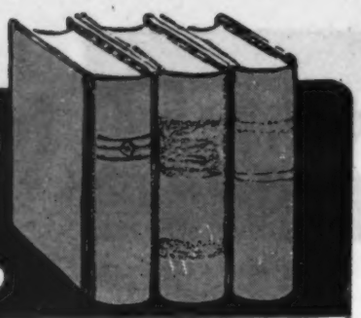

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per hour space velocity. Liquid yields are usually in excess of 95 percent, and most of them 97-99 percent. The best catalyst is alumina, the activation of which is maintained by impregnation with anhydrous hydrogen chloride. The life of this catalyst appears to be long if the burn-off is carefully controlled so that the catalyst is thoroughly cleaned but not sintered. The improvement of octane number approximates four units when a charge stock such as debutanized stabilized distillate is subjected to isomerization. Thermally cracked distillates are the best charge stocks because of their high olefin content and relatively low octane number. The octane numbers of many of the olefins in the gasoline boiling range are given in tabular form.

The Life of a Cobalt Catalyst for the Synthesis of Hydrocarbons at Atmospheric Pressure. C. C. HALL AND S. L. SMITH. *Jour. Soc. Chem. Ind.* **65** (1946) pp. 128-36.

Commercial operation of a process employing catalysis on an industrial scale largely depends on the period of time the catalyst can be maintained in an active condition. Published accounts covering prewar operations of Fischer-Tropsch plants in Germany make it appear that this period of operation was 4 to 5 months for the cobalt catalyst used in the synthesis of hydrocarbons at normal pressure. The manufacture and recovery of catalyst forms an important and expensive feature of the whole process. The work described in the author's paper shows that a cobalt catalyst supported on kieselguhr and containing thoria and magnesia as activators can be maintained in a high state of activity for periods of the order of 18 months if the catalyst is given intermittent treatment with hydrogen under various conditions. The temperature of the hydrogen treatment is in the synthesis range. Experiments indicated three processes leading to the deterioration of the catalyst, aside from poisoning caused by sulfur compounds in the synthesis gas.

The Calculation of Temperature Distribution in Catalytic Converters. L. M. GROSSMAN. *Trans. Amer. Inst. Chem. Engrs.* **42** (1946) pp. 535-51.

A problem of industrial importance is the calculation of the transfer of heat between a fluid stream and the granular solid through which it is flowing, and the distribution, with respect to time and position, of temperature in the fluid and in the solid. Applications come in the distillation and absorption column design, catalytic converters, regenerators, recuperators, blast furnaces, and the like. Finite difference and graphical procedures were applied in the analysis of temperature distribution for several ideal systems. Two cases were considered: (1) the transient temperature distribution in a cycling or batch process, in which heat transfer by conduction within the granular solid and fluid stream may be assumed zero; (2) the steady-state temperature distribution including heat transfer by conduction in the solid perpendicular to the direction of fluid flow. The stepwise graphical methods discussed offer the advantage of permitting consideration of the change of fluid and solid properties which are known but are arbitrary functions of temperature and position, and the satisfaction of boundary conditions that may

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Uncatalyzed Reaction of Natural Gas and Steam. A. S. Gordon. *Ind. & Eng. Chem.* **38** (1946) pp. 718-20.

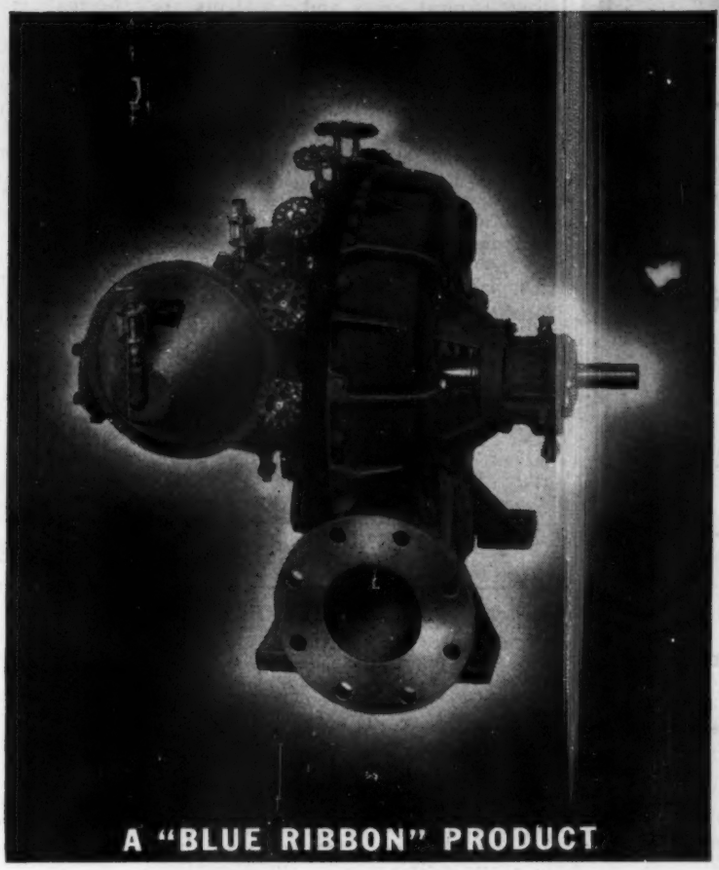
The catalyzed reaction between steam and natural gas has been studied by many investigators and is used industrially to produce a mixture of hydrogen and carbon monoxide at temperatures from 750° to 900° C. The authors believe there is an attractive industrial possibility of using cheap and efficient heat transfer in gas systems to effect the uncatalyzed natural gas-steam reaction. Accordingly, the uncatalyzed reaction of natural gas with steam was studied between 1225 and 1516° C. with steam-natural gas ratios of 1.5 and 5, and time of contact between 0.21 and 4.6 seconds. Even at high temperatures and long contact times, and steam-natural gas ratio of 5, there is some carbonization of the natural gas. The extent is shown to be a function of the ratio of steam to natural gas, and to vary erratically with temperature and time of contact. As the temperature is increased, the percentage of natural gas undecomposed in one pass through the furnace becomes less dependent on contact time until at about 1500° C. there is little or no effect. At any temperature the percentage of undecomposed gas shows little if any dependence on the ratio of steam to natural gas. The results of the work indicate that the design of pilot plants for the steam-natural gas reaction must make allowance for an indeterminate but appreciable amount of carbon deposition, for even at the highest temperature used in the tests, some carbon deposits.

Nomograph for Calculation of Over-all Heat Transfer Coefficients. H. J. Garber and W. Licht, Jr. *Ind. & Eng. Chem.* **38** (1946) pp. 757-59.

The authors present a nomograph for the calculation of over-all heat transfer coefficients. A typical problem involving the over-all heat transfer from steam to water through a copper wall is given. Three typical cases are included, one in which the wall is a flat sheet 0.5-inch thick, the second in which the wall is a pipe with 1-inch O.D. and 0.64-inch I.D., and again in which the wall is a sphere with 10 feet O.D. and 900 feet I.D. The nomograph is used in the solution of this problem, as well as the second in which the film coefficients are taken as having different values.

Optimum Water Requirements vs. Mean Temperature Difference in Single-Shell-Pass — Multi-Tube-Pass Condensers and Coolers. J. Meisler. *Trans. Amer. Inst. Chem. Engrs.* **42** (1946) pp. 553-66.

The author analyzes a variation of the cooling-water-outlet temperature in single-shell-pass multitube-pass condensers and coolers in terms of its effect on the overall logarithmic mean temperature difference in the unit. A limiting condition for a desirable optimum water rate is stated and a mathematical expression derived incorporating the above condition. A graphical solution of the latter expression is given. An expression for an absolute minimum water rate is developed and shown to be a



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limiting condition for exchanger operability. Typical cases and problems are considered. The author concludes that in condensers and coolers the most desirable design is to allow enough water to have the outlet temperature less than the corresponding shell fluid temperature. Where water is scarce, the water rate that will result in equal outlet temperatures for both streams is the minimum for which the engineer should aim. Any lower quantity will result in poor performance.

Displacement Capacities of Mixing Impellers. J. H. RUSHTON, D. E. MACK AND H. J. EVERETT. *Trans. Amer. Inst. Chem. Engrs.* **42** (1946) pp. 441-54.

A method is described for determining the flow of water caused by a propeller or turbine in a mixing tank. The method as applied to propellers gives results believed to be close to the true discharge capacity under conditions of normal mixing. Quantitative data on discharge capacities of impellers operating without casings and based upon direct experimental evidence do not appear to be in the literature. There is enough difference between the capacities of impellers operating in the free space conditions present in a mixing tank and impellers run in pump casings as to raise question as to how accurately pump impeller data can be applied to mixing impeller performance. The technique described is designed to obtain flow data under conditions as close as possible to those that exist in an actual mixing tank. Results are presented for marine-type square-pitched propellers from 4 to 12 inches in diameter. Adaptation of the technique to the flow from a flat bladed turbine impeller is described. Results are given for one diameter of turbine of a type commonly used in industrial operations.

Products: Properties and Utilization

Viscosity of Lubricating Oils at Low Temperatures. L. G. ZHERDEVA, Z. VOSSHINSKAYA AND O. FEDOSEEVA. *Petroleum* (London) **9** (1946) pp. 76-7.

When oils containing 2 percent of paraffin were cooled to —15° C., paraffin wax crystallized with formation of a crystal latice and possibly with a change in the viscous properties of the molecules composing the oil. Formation of a crystal latice at temperatures above the pour point, causes an anomalous viscosity dependent on such factors as thermal history of the oil, stirring, etc. Stirring, prior to measurement of viscosity, can change viscosity as much as 20-25 times. No solid paraffins could be separated from the low-pour-point transformer oils by filtration. The change in viscosity of oils at low temperatures depends largely upon structure. Synthetic lubricating oils have exceptional mobility at low temperatures because of structure. Knowledge of the usual constants of an oil is insufficient to indicate its suitability as a lubricant for cold starting. The change of viscosity with pressure has practical significance, since the viscosity/temperature coefficient of oil decreases with increasing pressure. Low-viscosity oils have a more favorable viscosity-temperature curve, than more viscous oils in the low-temperature region. Oils of cyclic structure are more sensitive with regard to changes in viscosity caused by a pressure than are oils of straight-chain structure. The addition of a synthetic oil to a low-grade petroleum oil greatly improves the fundamental characteristics of the latter.

Silicones as New Engineering Materials. W. R. COLLINGS. *Trans. Amer. Inst. Chem. Engrs.* **42** (1946) pp. 455-71.

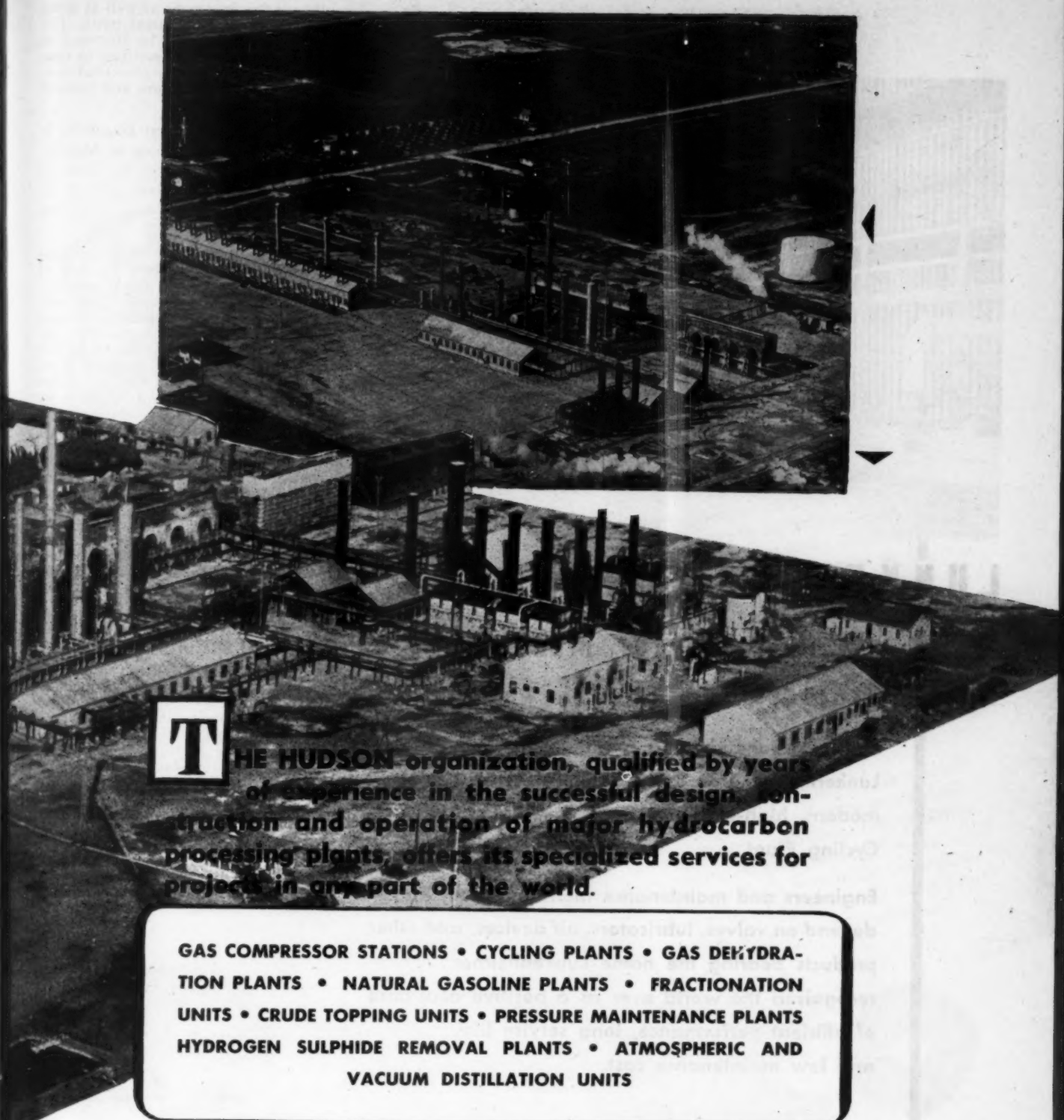
A series of hydraulic fluids, lubricating fluids and diffusion pump oils based on silicones have been developed and are in commercial production. Silicone lubricating greases for high-temperature bearings, conveyors, plug valves, and other special uses are being manufactured. Bonding resins for laminating Fiberglas or asbestos cloth produces boards and tubes that are moisture, acid, and heat resistant. Protective coatings having exceptional resistance to most destructive elements have been produced but they require relatively high curing temperatures. Silicone insulated electrical equipment offers the possibility of greatly decreasing the fire hazard in chemical plants, and of preventing motor and other electrical failures under conditions of high temperature, high humidity, or corrosive materials. Silicone rubber having exceptional resistance to deterioration by heat, fire, and moisture is finding increased use in electrical insulation, gaskets and other similar engineering applications. The chemistry of the silicone products is discussed and compared to the chemistry of carbon.

Small Glass Center-Tube Fractionating Column. E. A. NARAGON AND C. J. LEWIS. *Ind. & Eng. Chem.*, Anal. Ed. **18** (1946) pp. 448-50.

The authors note that a fractionating column capable of efficiently separating small quantities of close-boiling liquids is of great laboratory utility, and that to be of practical value such a column must have a low operating holdup. An improved small glass center-tube fractionating column that can be easily reproduced is described, and construction details are given. The fractionating section, which is surrounded by a silvered vacuum jacket, is 30.5 cm. long and is made of an 8-mm. inside diameter outer tube and a 6.5-mm. outside diameter center tube. A distributor is used above the fractionating section in order to distribute the reflux from the head to the outer tube and to the center tube. An automatic take-off valve is used. The column functions in such manner as to be equivalent to 75 theoretical plates with an H.E.T.P. of less than 0.4 cm. Data are given on efficiency tests and on the distillation of the known mixture of n-heptane and on methyl cyclohexane.

Preparation of Synthetic Samples of Low-Boiling Hydrocarbons. R. H. BUSEY, G. L. BARTHAUER AND A. V. METLER. *Ind. & Eng. Chem.*, Anal. Ed. **18** (1946) pp. 407-11.

The design and operation of an apparatus for making blends of low-boiling hydrocarbons is described. Standard samples containing any number of components can be prepared quickly, and with a degree of accuracy higher than that of present methods of analysis. Sketches are included showing the as-



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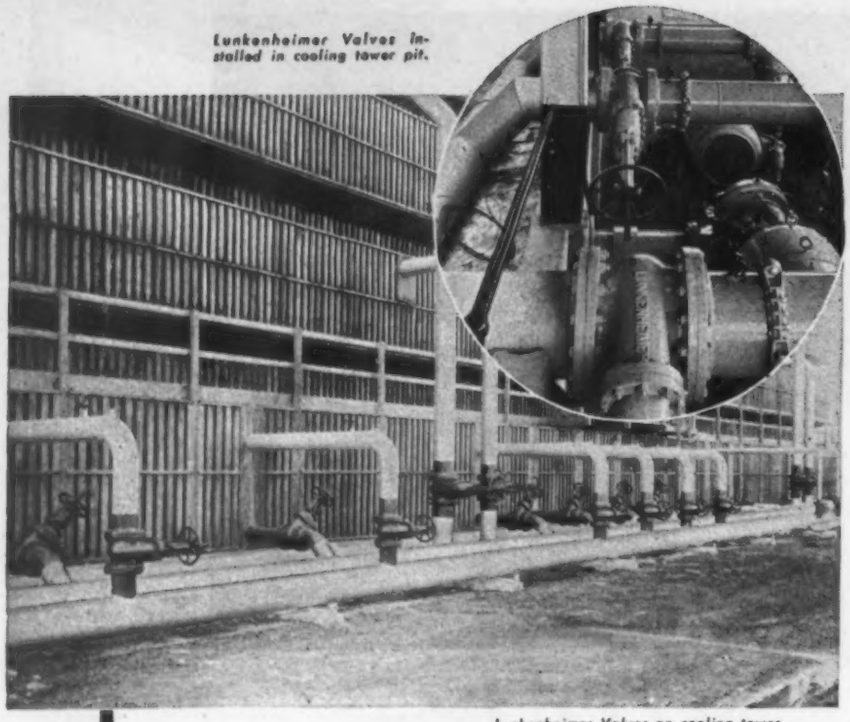

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sembly of the apparatus as well as some of its important component parts. The operating procedure to be followed in sample preparation is described in considerable detail. Data are presented covering samples in the butane and pentane range.

Determination of Alkyl Disulfides in the Absence and Presence of Mercaptans. I. M. KOLTHOFF, D. R. MAY, P. MORGAN, H. A. LAITINEN AND A. S. O'BRIEN. *Ind. & Eng. Chem.*, Anal. Ed. **18** (1946) pp. 442-44.

In the method described disulfides are reduced to mercaptans by use of amalgamated zinc and titrated amperometrically with silver nitrate. If the mercaptans are present in relatively small amounts in the original sample, the disulfide is determined by titrating portions of the solution before and after reduction, thus obtaining the mercaptan produced by reduction of disulfide by difference. The disulfide, when present in small amounts, the mercaptan in the original sample is removed by precipitation as silver mercaptide. The disulfide then is reduced for determination by titration. The accuracy of the method is limited by the degree of reduction of the disulfide by the amalgamated zinc. The results are about 5 percent low if the amount of disulfide is in the optimum range. The results are reproducible to 2 percent.

Lancaster Joins Magnolia Technical Department

George H. Lancaster has joined the staff of the technical department of Magnolia Petroleum Company, Beaumont, Texas, as automotive engineer. He recently returned to civilian life following duty in the Navy during which he was engaged in research and development activities of the bureau of ordnance on aircraft rockets. He formerly was employed in aircraft engine-test and development work with Wright Aeronautical Corporation and with Jacobs Aircraft Engine Company. He had been associated previously with the fuels and lubricants activity of the automotive laboratory of Tide Water Associated Oil Company.

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Patents Pertaining to Petroleum Refining

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REFINING

U.S.P. 2,402,587. Chemical Process. A. M. Alvarado, W. A. Lazier and J. H. Werntz to E. I. duPont de Nemours & Company.

Mono-olefin hydrocarbons containing at least 6 C atoms and preferably more than 8 C atoms are converted to sulphonic acids having valuable properties as surface-active agents by reacting the olefin at elevated temperatures (125-200°C.) and pressure with free sulfur (at least 3 mols per mol of hydrocarbon) to produce a sulfurized olefin, hydrogenating the product at 125-200°C. under 100-300 atmospheres pressure in the presence of a sulfacid hydrogenation catalyst to give a product consisting mainly of monothiol, and oxidizing the product with 70 percent nitric acid solution at 60-100°C.

U.S.P. 2,402,685. Chemical Process. F. K. Signaigo to E. I. duPont de Nemours & Company.

Aryl sulfides are produced by reacting an aromatic hydrocarbon with a sulfur halide in the presence of a limited amount of a sulfurization catalyst (not exceeding 10 percent and preferably not more than 5 percent by weight of the reactants). Napthalene and sulfur monochloride are reacted at 50-140° C. in the presence of zinc chloride.

U.S.P. 2,402,804. Treatment of Hydrocarbon Oils. V. L. Chechot to The Atlantic Refining Company.

Mineral white oils are produced by a two-stage treatment of the acid-refined oil with a decolorizing adsorbent, consisting of natural or synthetic metal silicate, each stage of the treatment being carried out at a different temperature. With finely divided adsorbents, temperatures of 230-260 and 200-210°F. are used for the two stages while with granular adsorbents the temperature ranges are 210-430° and 80-150°F.

U.S.P. 2,402,863. Blended Gasoline. H. H. Zuidema and J. E. Mital to Shell Development Company.

The presence of cyclic olefins, preferably containing a C_4 cyclo-olefin ring, in amounts up to 10 percent in gasolines tends to stabilize the gasoline against oxidation. A good effect is obtained by the presence of ½ percent of cyclo-olefin.

U.S.P. 2,402,954. Separation of Isomeric Paraffins. A. W. Francis to Socony-Vacuum Oil Company.

The separation of isomeric paraffins of a narrow boiling range can be effected by extracting with a cyclic organic solvent reagent which is miscible with or at least fairly soluble in water, relatively poor in selectivity for aromatic hydrocarbons and has a high dielectric constant. Such solvents, which can be used for instance to separate triptane from its isomers, are a.o. methanol, carbitol, ethyl sulfate, methyl cellosolve, acctic anhydride, acetonitrile, methyl carbitol, ethylene chlorhydrin and nitro methane.

U.S.P. 2,402,978. Process for the Chlorination of Saturated Hydrocarbons and Recovering Hydrochloric Acid. G. L. Allen, Jr., R. Heitz and G. Henderson to The Dow Chemical Company.

A gaseous mixture of HCl and at least one chloromethane is separated by scrubbing with a solvent to remove chloroform and carbon tetrachloride and provide a first gas fraction containing HCl. The solvent from the scrubbing step is heated to drive off methylchloride and HCl as a second gas fraction which is scrubbed with water to remove HCl and form a weak HCl solution. The first gas fraction is then scrubbed with this weak HCl solution to bring up the acid to commercial strength.

U.S.P. 2,403,054. Separation of Conjugated Diolefin Isomers. D. Craig to The B. F. Goodrich Company.

A mixture of piperylene isomers is reacted with SO_2 in such a manner that the low-boiling isomer reacts to form monomeric piperylene sulfone, but the high-boiling isomer remains substantially unreacted. The mixture of high-boiling isomer and SO_2 is then separated from the sulfone, the high-boiling piperylene and the SO_2 are separated and the sulfone is decomposed to form a mixture of low-boiling piperylene and SO_2 from which the low-boiling piperylene is isolated.

U.S.P. 2,403,088. Method of Treating Oils. K. M. Laing to Aluminum Company of America.

The steam emulsibility number of an oil is reduced by treating it in a conventional manner with an adsorbent consisting of fuller's earth and alumina in a ratio of not less than 1:1 and not more than 1:10. The alumina is prepared by heating a substance selected from the group consisting of aluminum hydrates and alumina in gelatinous form within the range of 250-800°C.

U.S.P. 2,403,127. Process for The Manufacture of Polynuclear Aromatic Hydrocarbons. J. E. Schulze.

Naphthalene is purified by comminuting it to make it permeable to a liquid extraction medium, and extracting impurities at 40-100°F. with a liquid medium containing from 80-95 percent of petroleum naphtha of a boiling range of 100-275°F. and from 5-20 percent of an aliphatic alcohol, ester or aromatic hydrocarbon of a boiling range of 100-275°F.

U.S.P. 2,403,485. Separation of Aromatic Hydrocarbons from Hydrocarbon Mixtures. H. V. Atwell and DuBois Eastman to The Texas Company.

Aromatic hydrocarbons are separated from mixtures containing them by extraction with water or with a solvent consisting essentially of water at a temperature considerably above the normal boiling point of water (475-525°F.) and under a pressure sufficient to maintain the solvent and hydrocarbons substantially in the liquid phase during extraction. A solvent and a hydrocarbon phase are formed and separated under pressure.

CRACKING AND REFORMING

U.S.P. 2,402,277. Manufacture of Diolefins. F. E. Frey to Phillips Petroleum Company.

Trimethylethylene is converted to isoprene by pyrolysis in a reaction zone at 750-850°C. at substantially atmospheric pressure for a period of time within the limits given by the equation $\log t = 13350 - (13.65 \pm 0.31)$ where t $\log t = 13350 - \frac{(13.65 \pm 0.31)}{T}$ where t is the reaction time in seconds and T is the temperature in degree Kelvin.

U.S.P. 2,403,268. Fuels for Aircraft Engines. G. H. B. Davis, W. J. Sweeney and W. A. Herbst to Standard Oil Development Company.

A fuel of satisfactory performance characteristics for supercharged aviation engines operating above 200 psi is produced by catalytically cracking a petroleum stock under conditions to produce a highly unsaturated gasoline and separating the gasoline into three fractions, viz.: (1) boiling below 100°F. and composed of C_4 hydrocarbons, (2) boiling between 100-140°F., and (3) boiling between 200-300°F. The first fraction is converted to iso-paraffins of 7-8 C atoms by alkylation, the second fraction is hydrogenated and an aromatic concentrate is extracted from the third fraction. A blend consisting of 56 percent of the products from the first, 19 percent of those of the second fraction and 25 percent by volume of the aromatic extract is prepared.

U.S.P. 2,403,281. Manufacture of Cyclopentanes. R. M. Hill and H. G. Codet to Standard Oil Development Company.

Alkylated cyclopentanes are dealkylated either by thermal treatment at 900-980°F. under 1000-5000 psi pressure in the presence of added hydrogen or by treatment in the presence of a catalyst containing a major proportion of magnesium oxide and minor proportions of copper oxide and manganese dioxide.

ISOMERIZATION

U.S.P. 2,402,807. Isomerization of Hydrocarbons. G. Egloff to Universal Oil Products Company.

N-heptane is isomerized to triptane by contacting it in the presence of a hydrogen halide with a metal halide catalyst at 0-75° C. In a primary stage substantial yields of methyl hexanes

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are produced which are separated and subjected in a secondary stage to contact with a metal halide catalyst in the presence of hydrogen halide at —25 to 50°C. Dimethylpentanes are separated from the products and contacted in a third stage with the catalyst at a temperature below —35° C. for the production of triptane.

U.S.P. 2,402,817. Isomerization of Hydrocarbons. J. O. Iverson to Universal Oil Products Company.

Paraffin hydrocarbons are isomerized with a metal halide catalyst in the presence of a hydrogen halide promoter. A hydrocarbon product containing metal halide and hydrogen halide is withdrawn from the reaction zone and contacted with a solid adsorbent (such as clay) at a temperature lower than that of the reaction zone. Metal halid is removed by the adsorbent and the effluent is fractionated to separate hydrogen halide from the hydrocarbons.

U.S.P. 2,402,818. Isomerization of Saturated Hydrocarbons. J. O. Iverson to Universal Oil Products Company.

The effluent from a metal halide isomerization zone is separated into a metal halide sludge and a hydrocarbon mixture containing free metal halide and hydrogen halide. The hydrocarbon mixture is fractionated to separate a vaporous overhead product containing isomerized hydrocarbons and hydrogen halide from a bottoms product containing a solution of free metal halide in hydrocarbon liquid, which is returned to the reaction zone.

U.S.P. 2,402,830. Conversion of Hydrocarbons. F. J. Moore to The Texas Company.

Saturated hydrocarbons are isomerized under a hydrogen pressure ranging from above atmospheric to 300 psi in the presence of a hydrogen halide promoter and a liquid catalyst body of specific gravity 1.3 to 1.4 prepared by reacting a metal halide and particularly $AlCl_3$ with a saturated paraffin hydrocarbon in the presence of both a small amount of CO and a small amount of MCl.

U.S.P. 2,402,858. Isomerizing Hydrocarbons. H. R. Warrick to The Texas Company.

Feed hydrocarbons such as n-pentane or a pentane-hexane fraction are subjecfed to contact with a metal halide-hydrocarbon complex catalyst in the presence of hydrogen halide at an isomerization temperature. After a substantial period of continuous flow, the catalyst is subjected to contact with a stream of lower-molecular-weight hydrocarbons such as n-butane, the catalyst being maintained at a temperature not greater tham the reaction temperature prevailing during treatment of higher-molecular-weight feed hydrocarbons and preferably at a somewhat lower temperature. The flow of lower-molecular-weight hydrocarbons is continued in the presence of hydrogen halide for a period of time sufficient to restore the activity of the complex catalyst. Normal operation then is resumed.

U.S.P. 2,403,181. Process for the Isomerization of Low-Boiling Saturated Hydrocarbons. J. P. Jones to Phillips Petroleum Company.

A process for the isomerization of n-butane to i-butane is described according to which a gaseous n-butane $AlCl_3$ mixture is passed through a zone containing an adsorptive mass until enough $AlCl_3$ has been deposited to give an active catalyst. N-butane and HCl are then passed through the zone in a reversed direction of flow and through a second zone containing an adsorptive mass. After catalyst activity declines, a n-butane-$AlCl_3$ mixture is passed first through the second and then through the first zone. $AlCl_3$ deposition occurs at 250-350°F., isomerization at 175-250°F. initially, at 275-325°F. after catalyst activity starts declining.

U.S.P. 2,403,279. Production of High-Octane-Number Fuels. A. P. Hewlett to Standard Oil Development Company.

Amylenes boiling at about 30-31°C. or a pentene-1-hexene-1 fraction from a product from the low-pressure vapor-phase cracking of higher-boiling hydrocarbons are contacted with bauxite at 750-850°F. to obtain higher boiling isomers which can be alkylated with i-butane. Before alykalation the isomerization product is purified by passage over clay or bauxite at 400-500°F. at 100 psi pressure.

U.S.P. 2,403,314. Hydrocarbon Isomerization Process. C. O. Tongberg to Standard Oil Development Company.

Some organic sulfur compounds are effective as promoters in the isomerization of n-paraffins in the presence of Friedel-Crafts type catalysts and hydrogen halide while others are harmful. Alkyl mono, di and tri sulfides are employed as promoters in amounts of 0.05-5 percent of the hydrocarbon.

U.S.P. 2,403,439. Process for Isomerizing Mono-Olefins. V. N. Ipatieff and G. S. Monroe to Universal Oil Products Company.

The double bond contained in olefin hydrocarbons such as butene-1, pentene-1, 2, 3-dimethyl butene-1, etc., can be shifted by the influence of catalysts comprising dilute aqueous solutions of strong non-oxidizing acids such as HCl or HBr or dilute acidic solutions of easily hydrolysed salts of such acids. Temperatures employed are 150-300°C. The pH of the catalyst solutions should be between 3 and 7.

ALKYLATION

U.S.P. 2,402,847. Alkylation of Aromatics with a Ferrous Chloride Catalyst. L. Schmerling and V. N. Ipatieff to Universal Oil Products Company.

Aromatic hydrocarbons and an alkylating agent selected from the group consisting of olefins and olefin-producing substances are contacted at 100-500°C. and under atmospheric —300 atmospheric pressure in the presence of a catalyst containing ferrous chloride and a carrier.

U.S.P. 2,402,243. Manufacture of Styrene. G. Egloff to Universal Oil Products Company.

Styrene is produced by subjecting benzene to inter-action with acetylene in the presence of supported catalysts of the Friedel-Crafts type, the reactions being brought about by passing mixtures of benzene and acetylene in regulated proportions (5:1 mol) over granular catalysts at relatively high rates so that short times of catalyst contact are made possible. A temperature of 0-150° C. is employed. Bauxite, precalcined at 500°C. and impregnated with 20 percent by weight of the composite of $AlCl_3$ is a suitable catalyst. The reaction time should not exceed 5 seconds.

U.S.P. 2,402,328. Production of Motor Fuel. E. C. Herthel to Sinclair Refining Company.

In the production of motor fuel by the alkylation of isobutane with butylenes in the presence of a catalyst, the steps are claimed consisting of bringing an excess of isobutane and the butylenes into intimate contact in the presence of a catalyst, separating the hydrocarbons from the catalyst, and subjecting the hydrocarbons to fractional distillation to recover therefrom the excess isobutane, introducing into the fractionating equipment in which the isobutane is separated a hydrocarbon stock containing isobutane, n-butane and butylenes derived from cracked stocks, operating said fractionating equipment so that a portion of butylenes having boiling points in between those of n-butane and i-butane is included in the i-butane-containing fraction and a portion in the bottoms fraction, and returning the overhead fraction to the reaction zone, the amount of butylenes in said overhead fraction being regulated so that the total quantity of butylenes supplied to the reaction zone is slightly greater than the total net quantity of i-butane supplied to said reaction zone, on a molar basis.

U.S.P. 2,403,013. Alkylation. H. R. Meadow to Socony-Vacuum Oil Company.

Tertiary aliphatic mercaptans, for example tertiary butyl mercaptan, will readily alkylate benzene or other aromatic compounds such as phenols when the reaction is carried out in the presence of as little as 0.0075 molecular weights of $AlCl_3$ per molecular weight of tertiary butyl mercaptan.

U.S.P. 2,403,124. Alkylation of Benzene. E. N. Rosenquist and R. W. Sudhoff to Monsanto Chemical Company.

Benzene containing from 10-30 percent by weight of polyethyl benzenes is reacted with ethylene or an ethylene-gaseous paraffin mixture in the presence of catalytic amounts of $AlCl_3$ and a constant concentration of hydrogen chloride amounting to from 0.15-0.30 percent by weight of the aromatic hydrocarbons. The HCl concentration is maintained by continuous addition of HCl in amounts which may range up to 20 percent of the introduced ethylene. The amount of polyethyl benzenes produced is equal to or less than that used in the feed. Up to 100 percent of the ethylene is reacted.

U.S.P. 2,403,342. Conversion of Hydrocarbons. R. B. Day to Universal Oil Products Company.

Isobutane and ethylene are alkylated in the presence of $AlCl_3$ while simultaneously isomerizing in the same reaction zone a normal paraffin of at least 5 C atoms.

U.S.P. 2,403,501. Alkylation Process and Products Produced Thereby. L. A. Clarke to The Texas Company.

A debutanized over-all alkylate from HF catalyzed alkylation of isobutane with an isobutylene alkylating agent substantially free from other olefin alkylating agents is claimed, which is characterized by an octane content of

In December, 1945 a UNISOL Mercaptan Extraction Unit was put into operation replacing a caustic mercaptan extraction unit and doctor sweetening unit at the Big Spring, Texas refinery of Cosden Petroleum Corporation. A careful full scale plant comparison between UNISOL treatment and caustic and doctor treatment was recently completed.

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Octane numbers and lead susceptibility

Treatment	Caustic Washed & Doctor Sweetened Octane No.	UNISOL Treated Octane No.
	66.6	68.3
Clear	71.1	73.1
+1 cc.	73.3	75.3
+2 cc.	74.6	76.7
+3 cc.		

COST OF TWO OPERATIONS

(UNISOL Plant Designed for 5000 BSD. Comparison Based on Throughput of 3200 BCD*)

Treatment	Caustic Washing & Doctor Sweetening	UNISOL TREATING	SAVINGS ($ per calendar day)
T. E. L.	2.15	1.15	$269 lead saving
cc./gal. 73.5 octane no.	579	310	
$/CD (0.2¢/cc. T. E. L.)			
Direct operating costs	5.25	4.20	$34 saving on operating costs
¢/bbl. thruput	168	134	
$/CD			
Increased recovery		0.9	
Estimated gasoline loss, %	1.2	28.8	$22 saved through reduced gasoline loss
BCD	38.4	66	
$/CD	88		

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at least 80 percent by volume, a 2, 2, 4-trimethyl pentane content of at least 50 percent by volume, a clear CFRM octane number of at least 96, and an AFD-3C rating with the addition of 3 cc. of lead per gallon of iso-octane reference fuels S_1 plus at least 2 cc. of lead per gallon at fuel:air ratios of 0.06 to 0.11 with especially outstanding performance at the lean fuel:air ratios.

HYDROGENATION, DEHYDROGENATION, AROMATIZATION

U.S.P. 2,402,493. Hydrogenation Process. B. S. Greensfelder and W. H. Peterson to Shell Development Company.

Diolefins are hydrogenated to monoolefins to the substantial exclusion of the corresponding saturated product with a substantial molar excess of hydrogen in the presence of a nickel sulfide catalyst. Suitable catalyst are prepared by impregnating a carrier with nickel nitrate or nickelous formate and decomposing at 250-350°C. The oxide is reacted with hydrogen sulfide at 400°C. Hydrogenation is carried out at 100-400°C. with a liquid hourly space velocity of 0.5-12.

U.S.P. 2,402,686. Hydrogenation of Arylsulphides and Products Resulting. F. K. Signaigo to E. I. duPont de Nemours & Company.

Aryl sulphide and hydrogen are reacted at 75-300°C. and under 100-300 psi pressure in the presence of a sulfactive hydrogenation catalyst.

U.S.P. 2,402,854. Hydrocarbon Conversion. C. L. Thomas to Universal Oil Products Company.

Aromatic hydrocarbons are produced from aliphatic hydrocarbons containing 6-12 C Atoms by contact at 450-700°C and under 50-100 psi pressure with a catalyst comprising a composite of alumina and chromia (4-12 percent) promoted by minor amounts (0.1-10 percent) of an oxide of a metal selected from the group consisting of copper, cerium, thorium and manganese.

U.S.P. 2,403,052. Method for Effecting Catalytic Dehydrogenation Process. R. M. Cole and I. I. Shultz to Shell Development Company.

Water-sensitive heavy-metal oxide dehydrogenation catalysts such as chromia or molybdena are regenerated by periodically burning off carbonaceous materials with an oxygen-containing gas. A sulfur-free dehydrogenation product is obtained, contamination of the catalyst by iron and damage during regeneration by SO_2 generation are avoided by adding between 0.0006 and 0.05 percent of sulfur to the feed stock to be dehydrogenated and, prior to regeneration removing sulfur as hydrogen sulfide from the catalyst by reaction with water vapor.

U.S.P. 2,403,276. Chemical Process. A. D. Green to Standard Oil Development Company.

Butylene is converted to butadiene in the presence of a catalyst comprising a major proportion of magnesium oxide and minor proportions of iron oxide, copper oxide and potassium oxide by heating the butylene to about 1100°F., superheating steam to about 1800-2000° F., mixing the two in a hydrocarbon to steam ratio of about 1:12 and passing them through the reactor, permitting the butylene a residence time of about 0.01-0.1 seconds.

U.S.P. 2,403,288. Process for Hydrogenating Olefins. H. F. Kohler to Standard Oil Development Company.

The hydrogenation of olefins is carried out in a plurality of reactors. About twice the amount of hydrogen consumed in the process is charged to the first reactor and passed to subsequent reactors, while the olefin feed is split between the reactors. Saturated hydrocarbons are separated from the hydrogen stream between successive reactors.

POLYMERIZATION, CONVERSION

U.S.P. 2,402,875. Catalytic Conversion Process. P. W. Cornell to Jasco, Inc.

A process for the catalytic conversion of hydrocarbons which comprises forming a mixture of hydrocarbons to be converted and solid conversion catalyst in finely divided state, passing said mixture upwardly through a reaction zone of predetermined cross-sectional area, thereafter passing the resulting mixture upwardly through a second zone having a cross-sectional area different than said first named zone, removing the reaction products and catalyst from the last named zone, separating the catalyst from the reaction products, regenerating a portion of the catalyst so separated, thereafter mixing a portion of the regenerated catalyst with the remaining unregenerated catalyst and returning said mixture to the second named reaction zone.

U.S.P. 2,403,107. Catalytic Processes. S. H. McAllister, J. Anderson and W. E. Ross to Shell Development Company.

The effluent from a catalytic conversion zone employing a solution of $AlCl_3$ in $SbCl_3$ as a catalyst, is passed to a separation zone prior to fractionation. In this zone the effluent is contacted with cool hydrocarbon gases, to condense entrained catalyst components which are withdrawn as liquid from the separation zone.

DESULFURIZATION

U.S.P. 2,402,799. Separation of Sulfur and Aromatic Compounds from Oil. G. B. Arnold and H. V. Atwell to The Texas Company.

Naphtha containing aliphatic, alicyclic, aromatic and sulfur bearing compounds of the non-mercaptan type is extracted repeatedly with 2-4 volumes per volume of naphtha of a solvent, consisting essentially of water, at 400-600°F. and under sufficient pressure to maintain liquid phase. Sulfur-bearing compounds are extracted first, aromatics later.

U.S.P. 2,402,893. Conversion of Hydrocarbon Oils. S. H. Hulse to Standard Oil Development Company.

A high-boiling crude-oil fraction containing a relatively high percentage of sulfur is first subjected to a mild thermal-cracking treatment. The vaporous products are then subjected to catalytic cracking. The gasoline fraction formed from the catalytic-cracking operation is of the same high quality as that obtained by direct catalytic cracking of the original high-sulfur oil and the trade gasoil distillate is materially lower in sulfur. The preliminary mild cracking reduces the percentage of sulfur contained in gasoline and gasoil fractions.

U.S.P. 2,403,524. Process for The Preparation of High-Grade Antiknock Motor Fuels. A. Hagemann to The Alien Property Custodian.

Olefins having from 3-5 C atoms are subjected to selective polymerization of the iso hydrocarbons present. The resulting product is treated with carbon monoxide and hydrogen under conditions (at least 300°C.) to form alcohols which are then dehydrated to alkenes and hydrogenated to high-octane-number alkanes.

CATALYST PREPARATION ACTIVATION AND REGENERATION

U.S.P. 2,402,126. Alkylation of Hydrocarbons. L. A. Clarke to The Texas Company.

When alkylating hydrocarbons with an $AlCl_3$ catalyst, the catalyst deteriorates rapidly due to the formation of gummy and sticky compounds which bind solid particles of catalyst together. This is avoided by maintaining the active metal halide suspended or dispersed in an agitated liquid body of preformed metal halide-hydrocarbon complex compounds. To some extent the active metal halide may be dissolved in the complex liquid. The ratio of complex liquid to active metal halide should be large.

U.S.P. 2,402,683. Hydrogenation Catalysts and Methods of Preparation. F. K. Signaigo to E. I. duPont de Nemours & Company.

The preparation of sulfactive hydrogenation catalysts is described. Finely divided metal in activated or pyrophoric form is treated with a sulfiding agent under conditions such that the heat of reaction is removed and the temperature of the reaction mass kept below 150°C.

U.S.P. 2,402,684. Hydrogenation Catalysts and Methods of Preparation. F. K. Signaigo to E. I. duPont de Nemours & Company.

Ferrous group metal sulfide catalysts are prepared by reacting a precipitated polysulfide of a metal of the iron group with hydrogen at 75-200°C. until no more hydrogen sulfide is evolved.

U.S.P. 2,402,740. Production of Styrenes. F. T. Doumani and R. F. Deering to Union Oil Company of California.

Reaction rate and life of catalysts in the dehydrogenation of ethyl benzene to styrene is increased by the use of catalysts composed of carbon containing certain metals such as nickel. The reaction is carried out at 800-1600°F. in the presence of a catalyst containing from 1-20 percent metallic nickel which may be prepared by impregnating a carrier with nickel salt solution, decomposing and treating the nickel oxide catalyst with ethylene under decomposition conditions to deposit carbon, followed by reduction with hydrogen.

HEAVY OILS AND WAXES

U.S.P. 2,402,288. Manufacture of Lubricating Oils. A. Lazar and J. C. Carter to Tide Water Associated Oil Company.

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THE PHOTO ABOVE, taken in the Carteret, N. J., shops of Foster Wheeler Corporation, shows drilling a pair of large Anaconda Muntz Metal Tube Plates.

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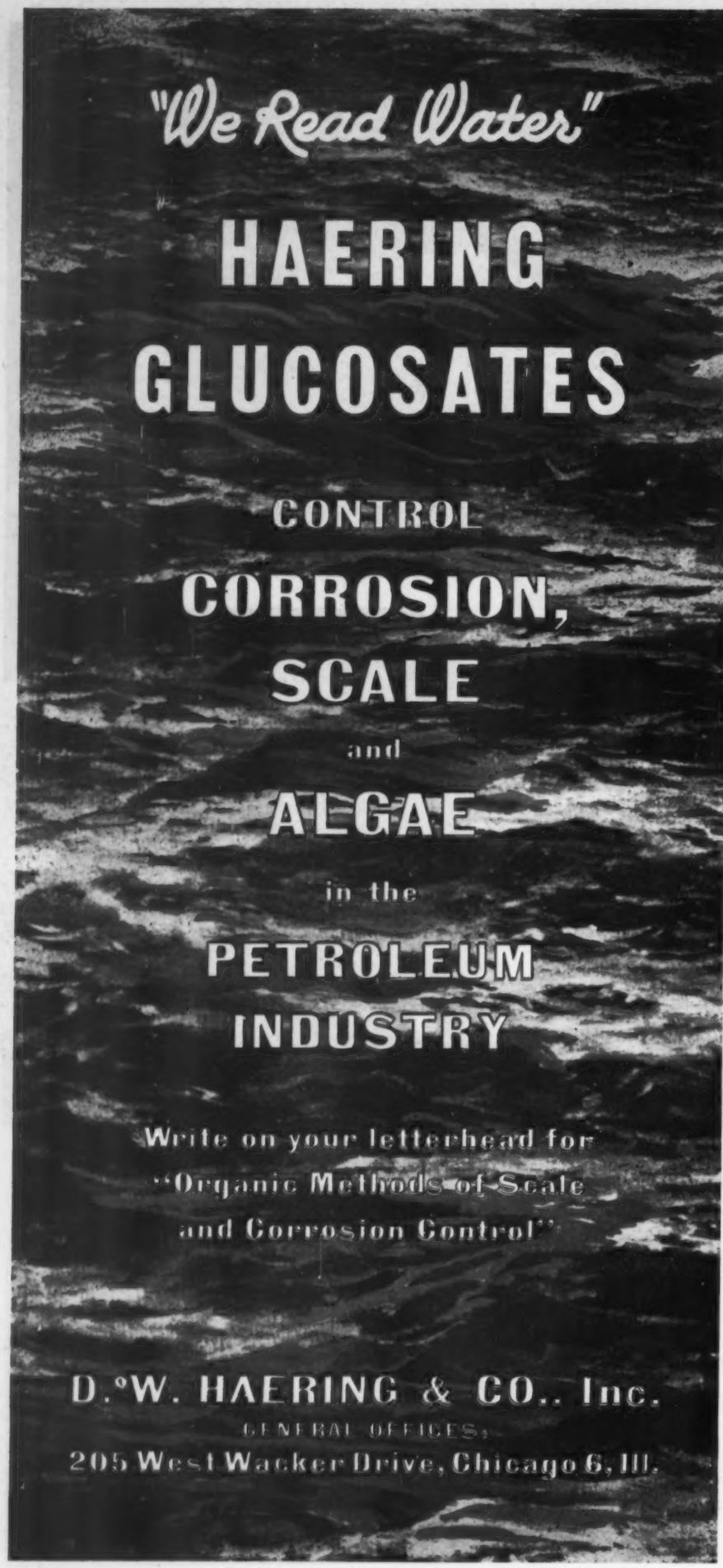

which is incorporated in a hydrocarbon lubricating oil is prepared by removing naphthenic acids and other acidic substances from a naphthene-base oil boiling above 375°F. at 2 mm. Hg. The purified oil is sulphonated, e.g. with concentrated sulfuric acid, spent sulphonating agent is removed and sulphonic acids are converted to polyvalent metal sulphonate free of alkali metal sulphonate. The amount of metal sulphonate in the finished oil should be in excess of 1 percent.

U.S.P. 2,402,427. Method of Treating Diesel Fuels. P. Miller and E. Lieber to Standard Oil Development Company.

The ignition quality of a liquid hydrocarbon diesel fuel is improved by contacting the fuel as it is being fed to the combustion zone with a mass of normally solid, partially soluble sulfur-free ignition promoter. The promoter is selected from the class of arylnitroso amines or alkyl metal nitrates.

U.S.P. 2,402,969. Lubricating Composition. R. F. McCleary to The Texas Company.

Detergent properties are incorporated into a lubricating oil by the addition of from 0.01 to 5 percent by weight of an oil-miscible aliphatic-ether-alcohol derivative of the phenolic compositions present in or derived from the oils extracted from the Anacadium genus of the Anacardiaceae family, such as cardanol.

U.S.P. 2,403,267. Diesel Fuels. G. H. B. Davis to Standard Oil Development Company.

The cloud point of diesel fuel base stocks having a boiling range of 300-800°F. is reduced by the addition of 5-10 percent of a soluble polymethacrylic acid ester of a mono hydroxy alcohol having about 4-12 C atoms.

U.S.P. 2,403,284. Phosphatidic Lubricant and Method of Making. J. J. Jacobs to American Lecithin Company.

The oxidation of mineral lubricating oils is inhibited by blending from 10-90 percent of the lubricating oil with a vegetable phosphatide and heating the mixture to 200-375°F. for 1-24 hours in the presence of air.

U.S.P. 2,403,453. Mineral Oil Composition. F. P. Otto to Socony-Vacuum Oil Company.

A mineral oil is stabilized against oxidation by the addition of from 0.1 to 1 weight percent of the reaction product of substantially one equivalent of a mono-hydroxy-aromatic compound, two equivalents of an aldehyde having more than 1 C atom and one equivalent of ammonia.

U.S.P. 2,403,474. Additive for Lubricants. J. D. Bartleson and F. Veach to The Standard Oil Company (Ohio).

Basic metal compounds react with the reaction products obtained from the reaction of certain amines with a sulfide or sulfides of phosphorus to give compositions containing the combined sulfide, amine and metal radicals or residues. These products are suitable lub oil additives when employed in amounts of 0.01-50 percent. The sulfide-amine reaction takes place at 100-400°F. The final product contains from 4-6 equivalents of zinc or barium per mol of phosphorus pentasulfide residue.

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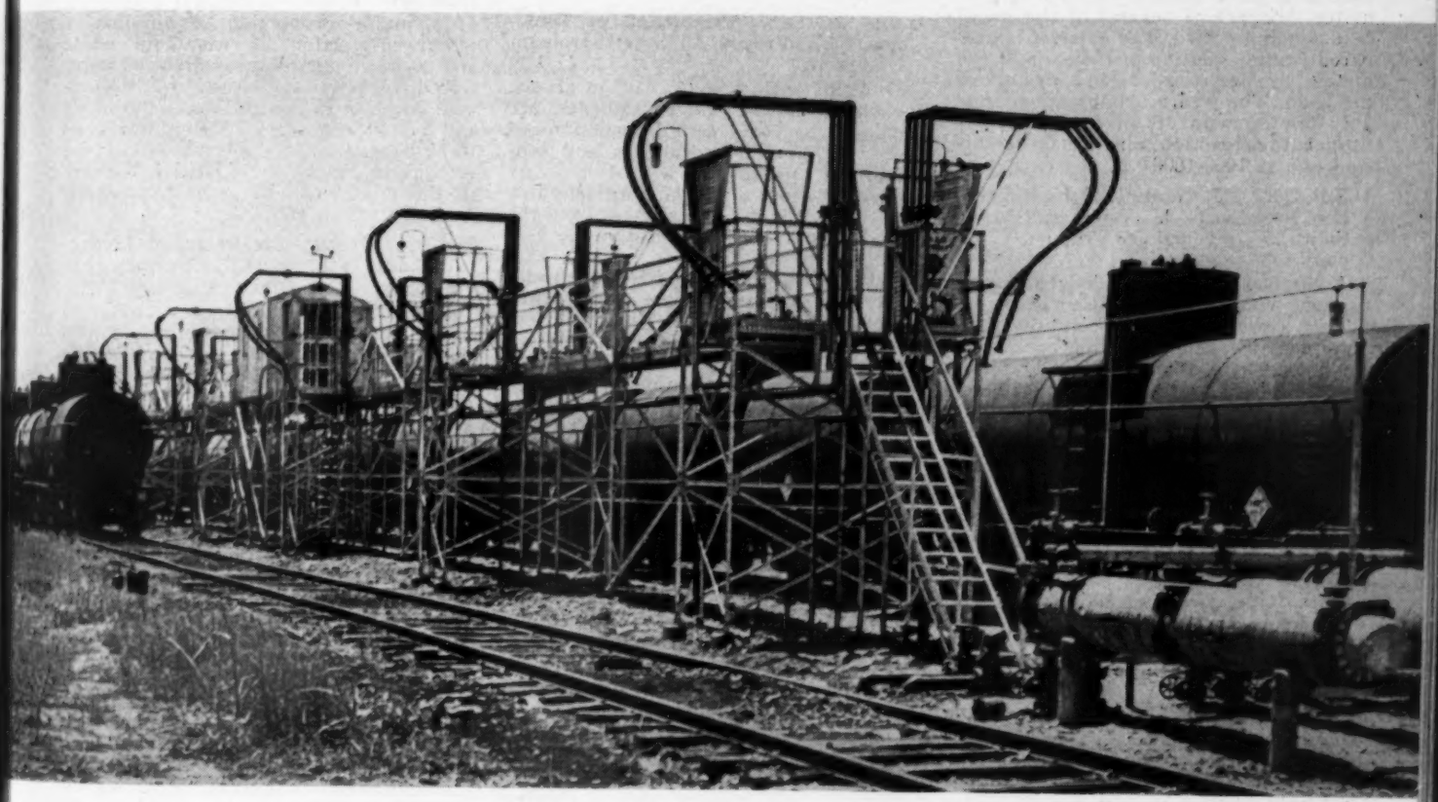

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DIGEST OF UNITED STATES PATENTS

On Dehydrogenation of Hydrocarbons

Compiled by HEINZ HEINEMANN

U.S.P. 2,370,797. Method for the Catalytic Dehydrogenation of Hydrocarbons. K. K. Kearby to Standard Oil Development Company.

Mono-olefins and aralkyl hydrocarbons with at least two carbon atoms are dehydrogenated by means of a catalyst comprising a major proportion of ZnO and a minor proportion of Fe_2O_3, MnO_2, Cr_2O_3, Co_2O_3 or Ni_2O_3 and in the presence of a small amount of K_2O as promoter. Mixture of several of the metal oxides may be used. The catalyst is prepared from a solution of the salts of the metals by precipitation and drying of the solid. The hydrocarbons are preferably diluted with steam for the dehydrogenation reaction which may be carried out at 1000-1600° F.

U.S.P. 2,371,087. Catalytic Dehydrogenation Process. G. M. Webb and M. A. Smith to Universal Oil Products Company.

A catalyst for the dehydrogenation of organic compounds is prepared by adding a precipitant to a solution of an aluminum salt and a soluble compound of at least one other catlytically active metal oxide. The precipitant must be of a nature such that it will precipitate only aluminum hydroxide and not the other metal compound in solution. The solvent is evaporated and the mixture obtained is heated sufficiently to convert the soluble metal compound into catalytically active oxide of metal on alumina. The soluble sales of difficulty reducible metal oxides, such as molybdenum oxide, chromium oxide and vanadium oxide, are primarily used.

U.S.P. 2,371,355. Motor Fuel. W. B. Ross and K. Boldt to The Pure Oil Company.

High-antiknock gasoline with a well-balanced distillation range is obtained by first separating from crude oil a light gasoline and a heavy gasoline fraction. The light fraction is dehydrogenated by means of a dehydrogenating catalyst at about 752-1292° F., and the heavy fraction is hydroformed or isomerized by means of a hydroforming catalyst in the presence of hydrogen at elevated pressure and at a temperature of about 950-1050° F. The products thus obtained then are blended. Alternately the heavy gasoline fraction may be fractionated before further treatment into a fraction boiling in the range of about 235-425° F., leaving a still heavier fraction which is subjected to high-temperature cracking. Products thus obtained which are boiling within the range of 235-425° F. are then blended and subjected to isomerization as mentioned before.

U.S.P. 2,371,809. Process for the Dehydrogenation of Hydrocarbons. H. E. Drennan to Phillips Petroleum Company.

Diolefins are produced from corresponding mono-olefins with 4 to 6 C atoms by passing the latter admixed with an inert diluent gas over a brucite catalyst at 925-1400° F. and at a pressure of atmospheric to 50 psi. The quantity of diluent gas shall be such that a partial pressure of mono-olefins of the order of 0.2 to 1 atmosphere results. The flow rates shall be 1 to 10 liquid volumes of charge per volume of catalyst per hour. The unconverted mono-olefins are recycled. The active catalyst is prepared by heating natural granular brucite at 400-1500° F. in a stream of inert gas.

U.S.P. 2,371,817. Production of Diolefins. F. E. Frey to Phillips Petroleum Company.

Diolefins with 4 and 5 carbon atoms, such as butadiene and pentadienes, are obtained by the dehydrogenation of more saturated hydrocarbons and separation of the diolefins with 4 or 5 carbon atoms from accompanying dehydrogenation products. The effluent from the dehydrogenation is passed into the bottom part of an absorber while a lean selective absorption liquid is passed into the top part of the absorber. Unabsorbed gases are removed. The rich absorbent liquid leaving the absorber is passed through two strippers. In the first a less unsaturated hydrocarbon material is separated from a more unsaturated one and returned at least in part to the bottom part of the absorber. The more unsaturated hydrocarbon material is passed through the second stripper where a material predominantly comprising olefins is removed. The residual lean absorption liquid from the second stripper is recycled. A portion of the separated diolefin material is returned to the absorber.

U.S.P. 2,371,850. Process for Dehydrogenation of Hydrocarbons. W. A. Schulze and J. C. Hillyer to Phillips Petroleum Company.

Diolefins are produced from low-boiling aliphatic mono-olefins with at least 4 C atoms by passing the olefins at 1100-1300° F. into contact with a catalyst composed of a major proportion of an aluminum oxide (such as bauxite) and a minor proportion of barium or strontium hydroxide. The flow of the olefins is periodically interrupted to reactivate the catalyst by oxidation of deposited carbonaceous material. After this oxidation a gaseous paraffin hydrocarbon of 1-3 C atoms is paseed into contact with the catalyst at about dehydrogenation temperature for a time sufficient to substantially improve the activity of the catalyst which is then used again in the dehydrogenation reaction. The mono-olefins may be admixed with enough of an inert diluent to produce olefin partial pressures of about 0.1 to 0.5 atmosphere and the flow rates of the mixture over the catalyst may vary between 500 and 5000 gas volumes per volume of catalyst per hour.

U.S.P. 2,374,404. Catalytic Conversion of Hydrocarbons. J. E. Ahlberg to Universal Oil Products Company.

A catalyst for the dehydrogenation of hydrocarbons is described which consists of a major proportion of alumina and a minor proportion of chromium and zirconium oxide. The amount of chromium oxide is preferably greater than that of zirconium oxide.

U.S.P. 2,374,932. Dehydrogenation of Hydrocarbons. W. R. F. Guyer to Standard Oil Development Company.

Low-molecular-weight hydrocarbons are dehydrogenated by passing them at 1000-1600° F. over a catalyst consisting of a major proportion of alumina, a minor proportion of vanadium oxide and a small amount of silver. A preferred composition consists of 77-93 percent Al_2O_3, 5-15 percent vanadium oxide and 2-8 percent silver. The hydrocarbons to be treated may be diluted with an inert gas to reduce their partial pressure in the mixture to between 80 and 200 mm. Hg.

U.S.P. 2,375,021. Treatment of Hydrocarbons. J. C. Morrell to Universal Oil Products Company.

Aliphatic hydrocarbons are contacted under dehydrogenating conditions with an oxide of Ti, Zr, Ce, Hf or Th supported on an activated carrier comprising a calcined aluminum hydrate of high adsorptive capacity. A dehydrogenating temperature of 450-700° C., an absolute pressure of 0.25-7 atmosphere and a contact time of about 0.1 to 10 seconds are employed.

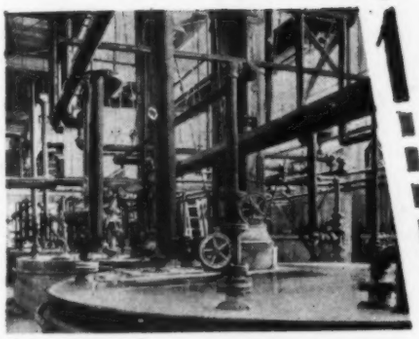
U.S.P. 2,376,059. Diolefin Production. J. P. Jones to Phillips Petroleum Company.

Diolefin hydrocarbons of 4 or 5 C atoms are produced from the corresponding more saturated hydrocarbons by dehydrogenating the latter while simultaneously forming minor amounts of lower-boiling unsaturated C_2 and C_3 hydrocarbons. The effluent from the dehydrogenation is separated into a lighter fraction comprising C_2 and C_3 hydrocarbons together with lighter gases and and a heavier fraction comprising C_3 and heavier hydrocarbons. C_3 and C_3 hydrocarbons are separated by absorption from the lighter fraction and then subjected to treatment at an elevated temperature and at low pressure to convert at least a part of them to the diolefins wih 4 or 5 C atoms with the simultaneous formation of more saturated hydrocarbons with the same number of C atoms. The effluent from the high temperature conversion is separated into a lighter fraction comprising C_3 and C_3 hydrocarbons together with lighter gases and a heavier fraction comprising C_3 and heavier hydrocarbons. Both lighter fractions are united prior to the subsequent separation of lighter gases. The heavier fraction from the dehydrogenation effluent is admixed with the heavier fraction from the effluent of the high-temperature conversion and the C_4 or C_5 diolefins are separated from the resulting mixture. The C_3 and C_3 hydrocarbons separated from this mixture are passed to the high-temperature conversion, while the more saturated C_4 or C_5 hy-

The 1, 2, 3 OF LICKING YOUR ACID PIPE PROBLEMS

The problem of controlling corrosion in the conveying of acids and other corrosive solutions is tough enough to begin with. But it is further complicated by such factors as elevated or fluctuating temperatures, pressure, vacuum and vibration or other mechanical stress of equipment.

That's why National Lead — as a result of its years of experience in every field handling corrosive solutions and gasses — makes available to you the wide variety of pipe described below. And why we offer you the first-hand "know how" gained by our technical staff from thousands of installations, to help you select the right pipe for every acid-conveying job.



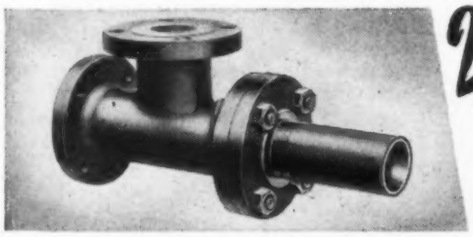
Making acid toe the line . . . Chemical Lead Pipe and Fittings employed in connection with a battery of digestors in a large industrial plant.

1 Lead Pipe. Widely used throughout industry is our standard Chemical Lead Pipe. Made of highly pure lead, it has a long record of successful applications. Like all of our lead pipe and pipe lining, it is fabricated by an extrusion process which assures uniform wall thickness, free flow and freedom from porous or defective spots.

Other highly useful types are made by alloying small amounts of other metals with the chemical lead.

For example, there is Tellurium Lead Pipe. This has added corrosion resistance at high temperatures and the ability to toughen and increase its tensile strength under mechanical stress. Still a third property — due in part to its capacity to work-harden — is a higher endurance limit and therefore greatly improved resistance to failure under vibratory stress.

Again, there is Antimonial Lead Pipe, made with a base of either chemical or Tellurium lead. When the antimony content is 6%, this insures a mechanical strength about twice that of chemical lead. And the greater hardness also means improved abrasion resistance — a real advantage where erosion is a problem. Recommended only for temperatures below 240° F.

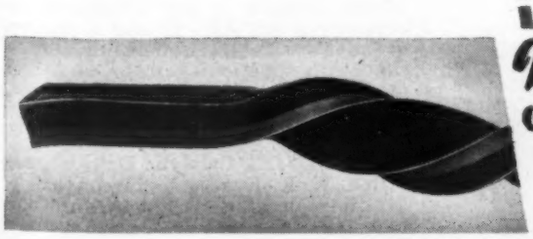


The strength of steel or iron, plus the corrosion resistance of lead . . . twin advantages of this "United" Lead-lined Pipe. Note the uniformity of the lead lining.

2 Cold Expanded Lead-Lined Pipe. Where the strength of iron or steel is needed — for example, to withstand pressure or other mechanical stress — our "United" Lead-lined Cast Iron or Steel Pipe has proved highly serviceable. It has the outer strength needed plus the inner resistance of its lead lining to corrosion.

The lining is fabricated in continuous lengths of uniform, seamless pipe, then cold expanded inside the iron or steel tube. This assures a tight fit and a lining smooth, uniform and free of defects.

Cold Expanded Lead-lined Pipe, as well as the "United Tubond" described below is made in flanged sections, with the lead lining carried over the face of the flange to give a positive lead-to-lead joint. Moreover all iron and steel pipe used meets the standards of the American Society of Testing Materials.



Once bonded . . . it stays bonded! A section of "Tubond" Lead-lined Steel Pipe, twisted to prove the inseparability of the metals. Steel and lead are married to stay.

3 "United Tubond" Lead-Lined Pipe. To do a job where equipment must stand extreme vibration, pressure, vacuum or temperature change — National Lead has developed "United Tubond" Lead-lined Pipe. The lead lining is bonded to the iron or steel tube by a special process, so permanent that there can be no separation or creep of the lining.

As shown in the illustration, the steel or iron can be violently twisted, but there will be no let-go of the lead.

A complete line of standard lead and lead-lined fittings is available for use with the various types of pipe. In addition, National Lead manufactures lead-covered pipe to individual specifications.

No matter whether you want a single valve . . . sheet lead for lining a tank . . . or a complete acid recovery plant . . . National Lead can supply you. You are invited to consult our technical staff on any equipment problem.

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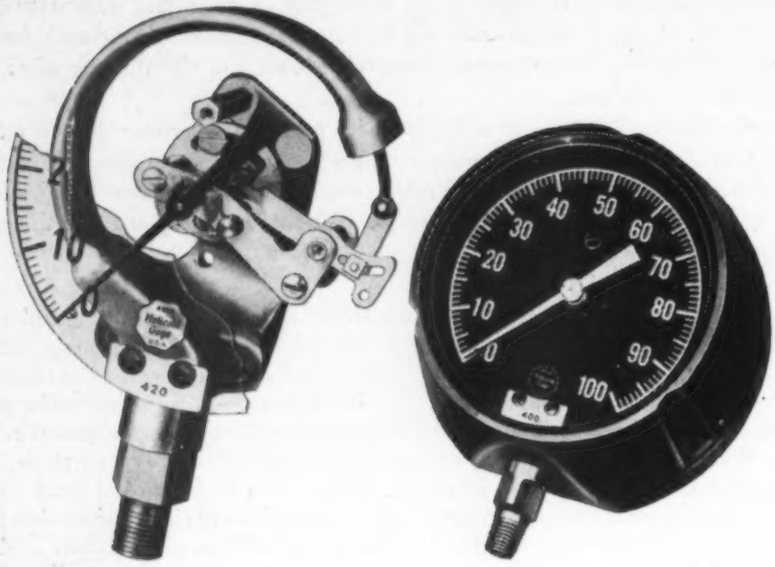

From the outside, a Helicoid Gage looks about the same as any other good pressure gage. Inside, it is unlike any other gage ever made.

What's the difference?

Well, for one thing, it has no spur gears. And the net result of that is that the Helicoid Gage will maintain its guaranteed accuracy ten times as long as a conventional-type gage.

That's only part of the story. The Helicoid Gage has at least four other exclusive superiorities.

If you use any quantity of pressure gages, servicing them is a considerable item of expense. We can show you how to save, both on gage costs and service costs.

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drocarbons of this mixture are passed to the dehydrogenation step.

U.S.P. 2,376,191. Chemical Process. B. E. Roetheli and W. G. Scharmann to Jasco, Inc.

Butadiene is produced continuously by discharging preheated butylene simultaneously with a dehydrogenation catalyst in the form of a fluidized powder into a reaction zone where they remain in contact at 950-1400° F. for 1-5 seconds. A quantity of cooled catalyst is added to the reaction mass at the end of the required reaction period to lower the temperature of the reaction mass below reaction temperature. Catalyst is separated from reaction products, regenerated and recycled.

U.S.P. 2,376,252. Oxide Catalyst in Dehydrogenation of Cymene. W. Hull to American Cyanamid & Chemical Corporation.

P-cymene is produced by passing vapors of a monocyclic terpene, such as dipentene, at dehydrogenation temperature (410-525° C.) over a catalyst composed of Cr_2O_3, CrO_3, or their mixtures and at least one of the oxides of copper, tin or zinc.

U.S.P. 2,376,309. Dehydrogenation of Menthane. J. K. Dixon to American Cyanamid & Chemical Corporation.

P-menthane is converted to dimethyl styrene by passing its vapor over a dehydrogenation catalyst composed of an oxide of chromium, molybdenum or vanadium, or mixtures thereof at a temperature of 450-600° C. for 2-20 seconds contact time.

U.S.P. 2,376,323. Process of Two-Stage Catalytic Dehydrogenation of Paraffin Hydrocarbons to Diolefins. R. G. Boatright, Jr., and W. C. Hewitt to Phillips Petroleum Company.

Paraffin hydrocarbons are dehydrogenated to diolefins by passing them through a first catalytic zone and then passing the olefin content of the effluent of the first zone through a second catalytic zone operating at higher temperature than the first zone.

U.S.P. 2,376,709. Production of Styrene. W. J. Mattox to Universal Oil Products Company.

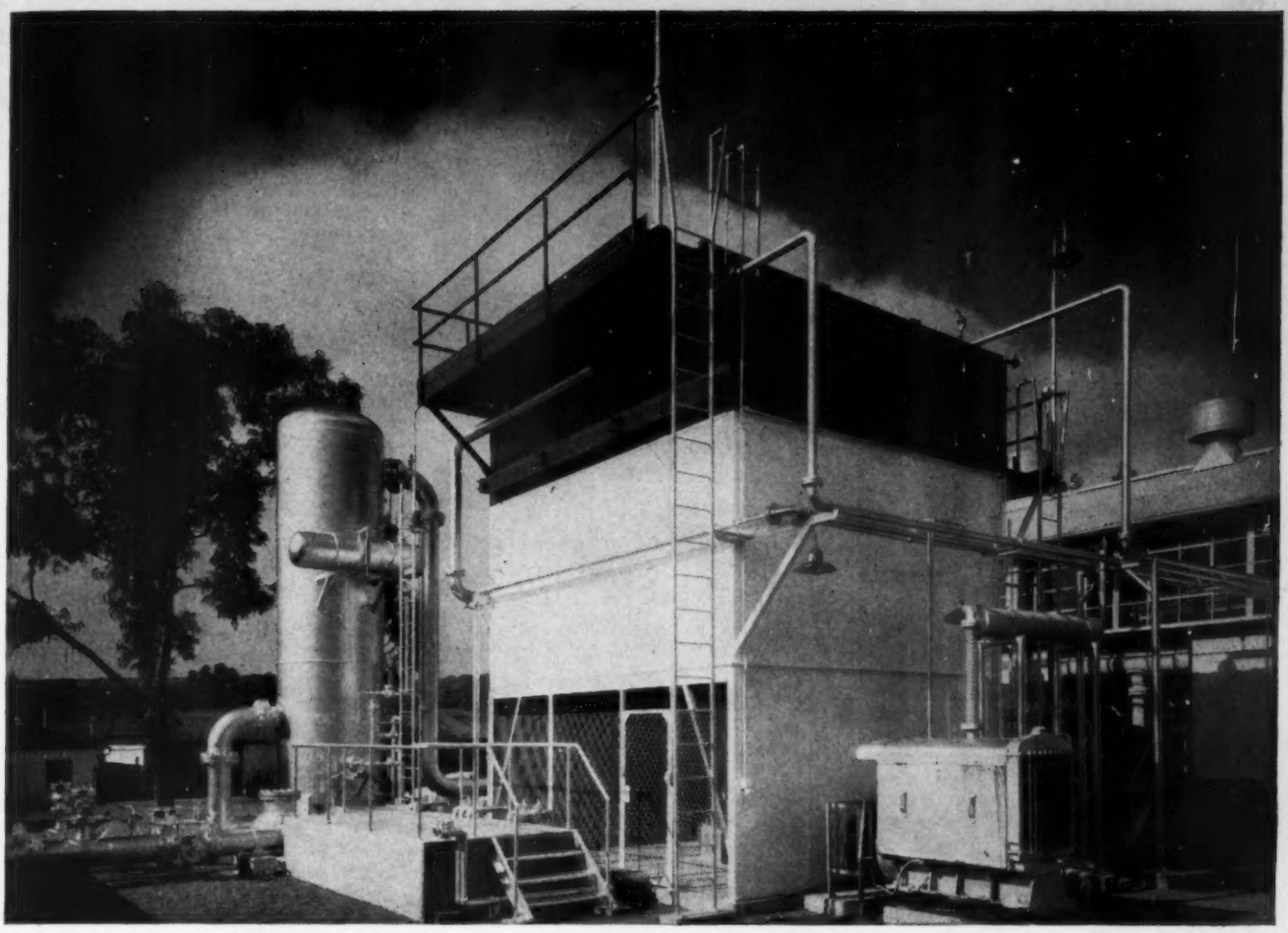
Styrene is produced from a mixture of ethyl benzene and xylene, for instance a suitable fraction of a straight-run naphtha obtained by the distillation of a Michigan crude, by contacting the mixture with a dehydrogenation catalyst under such conditions that the ethylbenzene is converted to styrene while the xylene serves as an inert diluent. A temperature of 450-700° C. and a pressure of about 0.25 atmosphere to slightly super-atmospheric is employed. Alumina promoted by a dehydrogenating metal oxide such as molybdenum oxide is the catalyst.

U.S.P. 2,377,083. Catalytic Dehydrogenation. K. K. Kearby to Jasco, Inc.

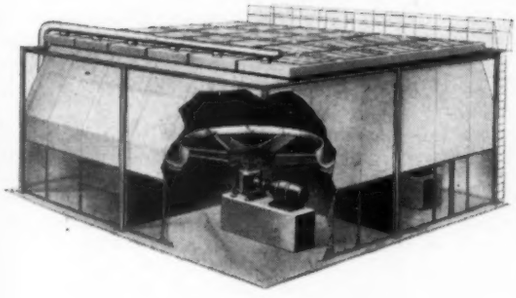
Low-molecular-weight hydrocarbons having 2-5 C atoms are dehydrogenated by passing them at a partial pressure of 40-250 mm. Hg and at a temperature of 1000-1600° F. over a catalyst consisting of a major part of MgO, a minor part of MnO_2 and a small amount of CuO. The catalyst contains e.g. 50-90 per cent MgO, 1-40 percent MnO_2 and 1-10 percent CuO.

U.S.P. 2,377,113. Dehydrogenation of Hydrocarbons. C. L. Thomas to Universal Oil Products Company.

Normally gaseous hydrocarbons are



FIN·FAN MEETS RIGID BUILDING REGULATIONS AND RENDERS TRIPLE SERVICE



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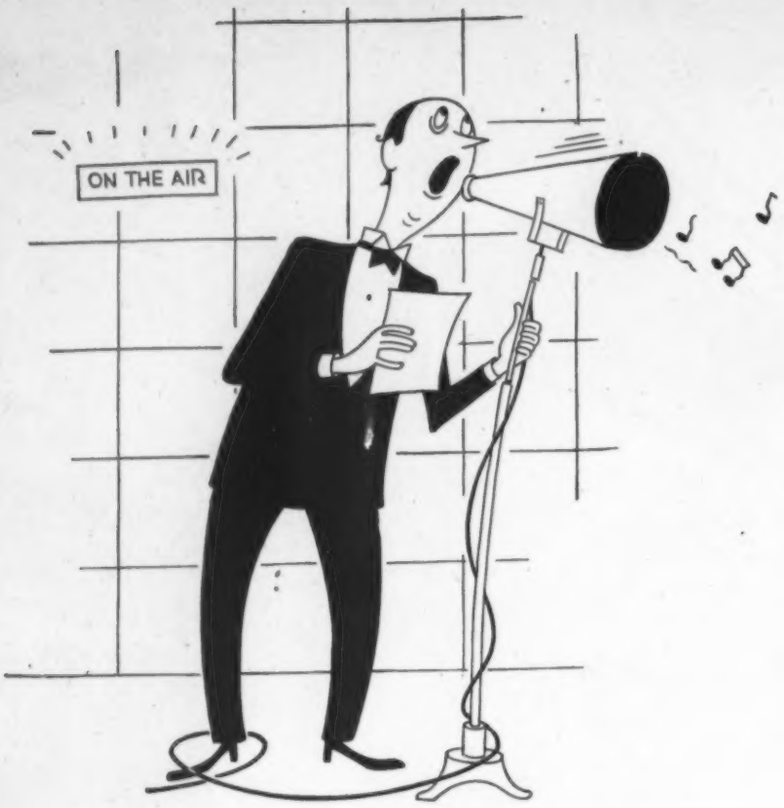

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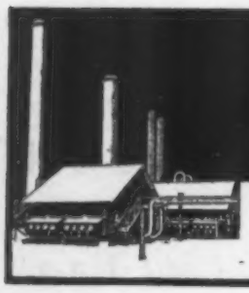

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dehydrogenated by contacting them with a catalyst composed of alumina and chromia and promoted by minor amount of an oxide of manganese. A temperature of 450-700° C. and a pressure not exceeding 50-100 psi is used in the dehydrogenation step.

U.S.P. 2,377,579. Process for Dehydrogenation of Hydrocarbons. W. A. Schulze to Phillips Petroleum Company.

Straight-chain olefins having at least 4 C atoms, such as butadiene, are prepared by dehydrogenation of the corresponding paraffin hydrocarbons in two steps, the saturated hydrocarbons being converted to the corresponding olefins, such as butene in the first step, and the resulting olefins being further dehydrogenated to the corresponding diolefins in the second step. Hydrogen is removed from the effluent of the first step and the olefin produced is admixed with at least an equal volume of diluent before passing it to the second step. The diluent comprises an olefin of lower molecular weight than the one produced in the first step and the paraffin corresponding to the olefin of the diluent. The olefin of the diluent, e.g. propylene, serves as hydrogen acceptor in the second step and is present in the diluent in excess of the concentration required for equilibrium with the corresponding paraffin (e.g. propane). Calcined bauxite is used as the catalyst in both steps.

U.S.P. 2,378,057. Treatment of Hydrocarbons. W. A. Yarnall to The Texas Company.

Gasoline hydrocarbons are dehydrogenated and cyclized by passing them at 400-700° C. over a zeolite catalyst in which the alkali metals have been replaced by a metal from the group of the left-hand column of Group VI of the periodic system, such as chromium, molybdenum, tungsten and uranium. For instance, n-heptane is contacted at 575° under atmospheric pressure for a period of 20 seconds with a molybdenum zeolite to produce a high yield of toluene.

U.S.P. 2,378,209. Process for Production of Aromatic Hydrocarbons. D. L. Fuller and B. S. Greensfelder to Shell Development Company.

A naphthenic gasoline fraction is first dehydrogenated by means of a metal sulfide catalyst, such as tungsten sulfide, then contacted with a molybdenum oxide catalyst and finally with a chromium oxide catalyst. (A temperature of 425-550° C. and a pressure of 10-70 atmosphere is maintained in the first dehydrogenation step.)

U.S.P. 2,378,649. Production of Butadiene. W. J. Mattox to Universal Oil Products Company.

C_4 olefins obtained by catalycically cracking an olefinic distillate or a polymer gasoline are dehydrogenated in the presence of a catalyst. The butadiene formed together with a minor quantity of iso-butene is separated from the latter and the unchanged C_4 olefins. The remaining mixture is again fed to the cracking step to isomerize the isobutene to normal butene.

U.S.P. 2,378,650. Manufacture of Butadiene. W. J. Mattox to Universal Oil Products Company.

This process is similar to that described in U. S. 2,378,649, but starts with a feed stock of normal C_4 hydrocarbons or normal butenes. The C_4 hydrocarbons (including isobutene) ob-

DISSOLVED OR ENTRAINED AIR

in circulating water—one of many factors governing condenser tube and heat exchanger tube performance—is presented in this discussion by Scovill engineers on selection of the proper tube alloy.

Fast-flowing rivers, rapidly changing tides, spray ponds and moving ships all create a condition causing entrainment of air. Furthermore, all raw water is saturated with dissolved air. Approximately 2.7 cu. ft. of air will dissolve in 1000 gal. of water — the quantity of air to saturate being dependent on the water pressure, composition and temperature. Any low-pressure areas in the circulating system permit the liberation of part of this dissolved air to ally itself with any free air in its erosive-corrosive attack on the tubes.

In actual operating conditions, where dissolved or entrained air in the circulating water is only one of the variables, tube life depends on the nature and/or extent of such other factors as: Velocity and turbulence of coolant . . . Chemical composition of circulating water . . . Temperatures inside and outside of tubes . . . Chemical or electro-chemical actions . . . Marine growths or other deposits . . . Sewage and wastes present . . . Development of protective film . . . Media surrounding tubes.

One of the principal objects of Condenser Tube Technical Research has been the development of alloys designed to give the user longest tube life and most trouble-free service. Where the major factor affecting tube life is the presence of dissolved or entrained air, satisfactory alloys include Cupro-Nickel, Aluminum Brass and Phosphorized Admiralty, each within its temperature limitations.

So many variables surround any condenser or heat exchanger tube application that each case must be treated as an individual problem.

Data accumulated during Scovill's continuous study of condenser and heat exchanger tube service conditions for every known application equip its Technical Service Department to offer sound guidance in tube alloy selection for existing or proposed installations.

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Scovill's *Service in Men* offers experienced engineering to help solve your tube selection and installation problems. *Service in Metals* develops and produces Scovill tube alloys that conform to your requirements. *Service in Manuals* consists of literature which gives you latest, authentic information on condenser and heat exchanger tubes. For a free copy of Condenser Tube Booklet, address Scovill Manufacturing Co., 15 Mill St., Waterbury 91, Conn. *Export Dept.* 405 Lexington Ave., New York 17, N. Y.

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tained in the dehydrogenation step in addition to butadiene are subjected to the action of an olefin isomerizing catalyst, e.g. precipitated silica, precipitated alumina and/or precipitated zirconia.

U.S.P. 2,378,651. Catalytic Process. M. P. Matuszak to Phillips Petroleum Company.

Hydrocarbons are subjected to endothermic catalytic dehydrogenation by passing them at a suitable temperature through a plurality of catalysts of different catalytic activities contained in an adiabiatic reaction chamber provided with temperature-controlling media. The catalysts are deposited in at least three concentric annular zones arranged successively and continuously relative to the inward or outward direction of flow of the hydrocarbon. One of the concentric zones contains a sufficient amount of a granular bauxite catalyst to decrease the temperature of the hydrocarbon to 1000-1070° F., another of the zones contains enough of a bauxite-supported chromium oxide catalyst to decrease the temperature of the hydrocarbon to 930° F., and a third of the zones contains enough black unglowed chromium oxide catalyst to decrease the temperature to 840° F.

U.S.P. 2,379,081. Catalytic Dehydrogenation. W. F. Huppke and Th. Vermeulen to Union Oil Company of California.

Gaseous or vaporous hydrocarbons are subjected at dehydrogenating temperatures, e.g., at 1100-1300° F., to the influence of molten boric oxide containing a small proportion of an activating metal oxide, such as ZnO, Fe_2O_3, CuO, VO or UO_2. The gases or vapors may be bubbled through the molten catalyst.

U.S.P. 2,379,172. Preparation of Catalyst and Method for Dehydrogenating Hydrocarbons with Same. M. P. Matuszak to Phillips Petroleum Company.

Hydrocarbons are contacted under dehydrogenation conditions with a gel-type metallic oxide catalyst prepared by adding a sol of alkali hydroxide to the solution of a salt of a metal capable of forming gelatinous hydroxides, first in such a quantity that a sol of colloidal solution of a basic salt is formed, about two thirds of the stoichiometrically equivalent amount of the alkali hydroxide being preferably used for this purpose. The sol is then added to such an amount of a dilute solution of the alkali hydroxide of not more than 0.1 normal concentration that the whole amount of the alkali hydroxide used in both stages slightly exceeds the stochiometric equivalent to the original metal salt to cause maximum precipitation of the metal hydroxide in a gelatinous and voluminous state. A mixed solution of chromic nitrate, zirconium nitrate and cupric nitrate or of aluminum nitrate and chromic nitrate may be treated in this manner with ammonium hydroxide.

U.S.P. 2,380,875. Catalytic Treatment of Hydrocarbons. W. A. Schultze to Phillips Petroleum Company.

1,3 butadiene is formed by dehydrogenation of a butene in the presence of an active bauxite-barium hydroxide catalyst. A stream composed of 1 mol butene and 2 mols steam, preheated to 1220° F. is passed to the catalyst zone at the rate of 1.0 liquid volumes of butene per volume of catalyst, and this

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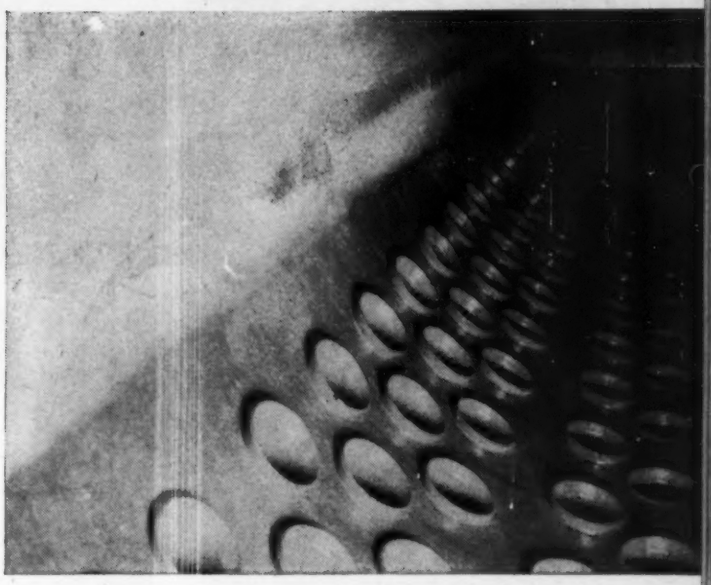

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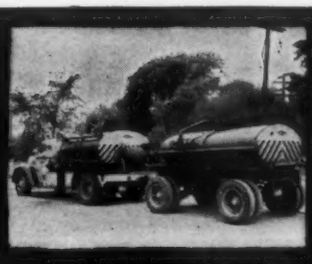

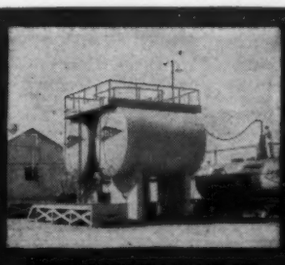

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stream is admixed with a second stream of steam of 740° F. in such a manner that the temperature of the mixed stream is approximately 1160° F. The flow rate of the second stream of steam is decreased progressively when the activity of the catalyst declines so that the temperature of the mixed stream at the inlet to the catalytic zone gradually increases from 1160 to 1220° F. The mol ratio of total diluent to hydrocarbon thus varies during the process from 3:1 to 2:1. An average conversion of 40 percent of the charge per pass was obtained and 50 percent of the converted material was butadiene.

U.S.P. 2,380,876. Process for the Dehydrogenation of Hydrocarbons. W. A. Schultze, J. C. Hillyer and H. E. Drennan to Phillips Petroleum Company.

Diolefins, such as butadiene are produced by dehydrogenation of monoolefins, such as butenes, by contacting the latter at dehydrogenation temperature (1100-1300° F.) with a catalyst consisting of a major proportion of bauxite and a minor proportion, (1 to 10 percent) of barium or strontium hydroxide. A pressure between 0.1 and 2 atmospheres and a space velocity of 1000-2500 may be employed.

U.S.P. 2,381,677. Process for Treating Catalysts. M. P. Matuszak to Phillips Petroleum Company.

In the catalytic dehydrogenation of hydrocarbons the induction period of the catalyst is reduced by heating the catalyst in an atmosphere containing free hydrogen. Subsequently the catalyst is flushed for not more than several minutes with a gas which contains a substantial proportion of a hydrocarbon that is more unsaturated than the charge stock. Thereafter the hydrocarbon to be dehydrogenated is passed over the flushed catalyst.

U.S.P. 2,381,691. Process for the Dehydrogenation of Hydrocarbons. W. A. Schulze and J. C. Hillyer to Phillips Petroleum Company.

N-butane is first treated with a dehydrogenating catalyst under such conditions that a substantial portion thereof is converted to butenes. The resulting mixture is fractionally distilled to separate a butene-1 fraction from a fraction which essentially comprises butene-2 and n-butane. The later fraction is recycled to the first dehydrogenation step, whereas the first fraction is treated in a second dehydrogenation step to convert part of the butene-1 to butadiene. The unconverted butene-1 is recycled to the second dehydrogenation step.

U.S.P. 2,381,692.

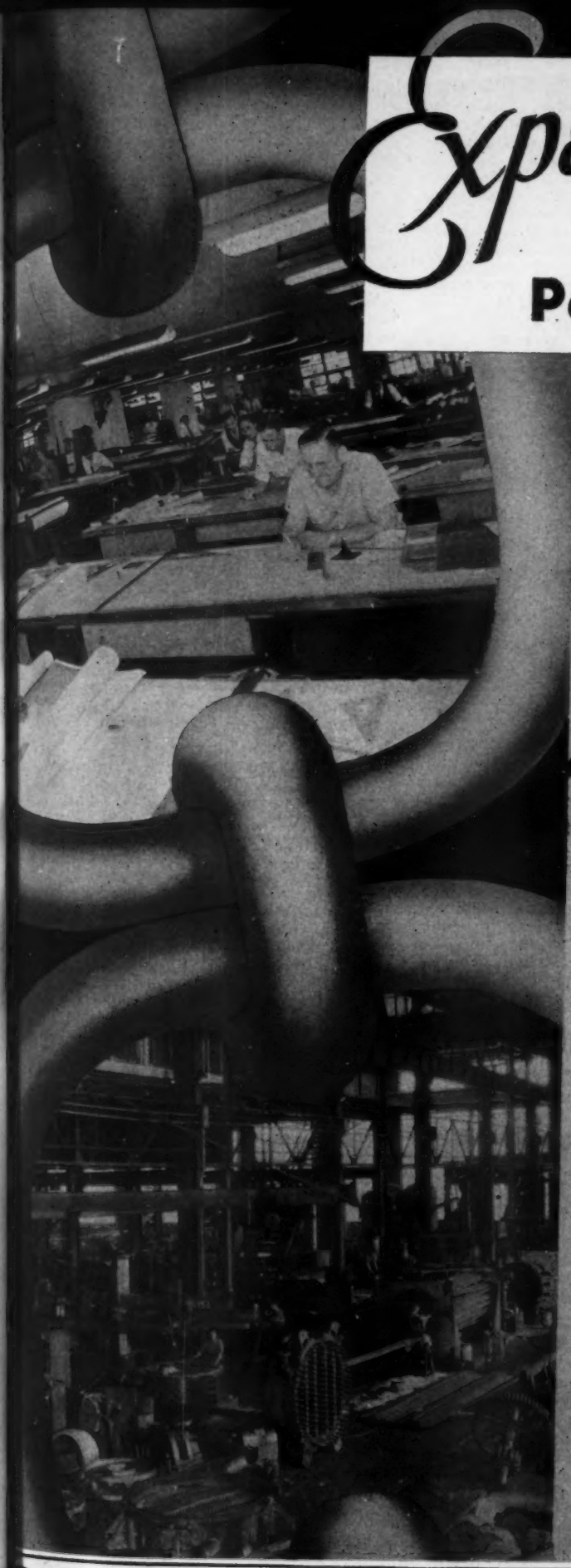
N-butane is continuously passed over a bauxite-metallic oxide dehydrogenation catalyst of relatively high catalytic activity at 950-1100° F. Otherwise this process resembles that of U. S. 2,381,691. The butene-1 formed in the first dehydrogenation step is in the second step passed over a less active bauxite catalyst at 1100-1300° F.

U.S.P. 2,381,693.

The invention concerns the process corresponding to U. S. P. 2,381,691 for the production of pentadiene from n-pentane.

U.S.P. 2,382,394. Treatment of Hydrocarbons. J. G. M. Bremmer, P. W. Reynolds and A. W. C. Taylor to Imperial Chemical Industries, Ltd.

Normally gaseous paraffin hydrocarbons or hydrocarbons with at least 3 C



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atoms are dehydrogenated in the presence of a catalyst which contains at least one of the following chromium compounds: potassium or sodium chromate, zinc chromate, zinc chromite, zinc dichromate, together with a product obtained by cautious thermal decomposition of a precipitate obtained from an aqueous solution containing an aluminum salt and ammonium dichromate, ammonium chromate or chromium trioxide, by adding thereto a precipitant consisting of NH_3, NH_4HCO_3 or $(NH_4)_2CO_3$ in an amount at least sufficient to give complete precipitation of the aluminum.

U.S.P. 2,383,643. Catalytic Dehydrogenation. S. T. Fulton and K. K. Kearby to Standard Oil Development Company.

Hydrocarbons are passed over a catalyst composed of a major proportion of MgO, a minor proportion of Fe_2O_3, and a small amount of a promoter formed by a fluoride of an alkali or an alkaline earth metal. Low-molecular-weight hydrocarbons, e.g. butene, are dehydrogenated in this way, preferably in the presence of steam.

U.S.P. 2,384,645. Process for Dehydrogenating Hydrocarbons. W. A. Schulze to Phillips Petroleum Company.

Butadiene and other straight-chain diolefins are produced from the corresponding mono-olefins by passing the charge stock admixed with C_3 hydrocarbons as a diluent in contact with a dehydrogenating catalyst at temperatures between 1000 and 1300° F. so that a considerable proportion of the olefin is converted into the corresponding diolefin. Immediately following this dehydrogenation a further quantity of the hydrocarbon diluent or of recycle liquid containing it is injected into the effluent of the dehydrogenation zone to reduce the temperature below the range of rapid diolefin polymerization. The diolefins formed are recovered from the cooled product and the diolefin-free product is recycled to the dehydrogenation zone. The formation of polymers and tar is greatly reduced and the yield of diolefin increased.

U.S.P. 2,384,984. Manufacture of Styrene and Ethylbenzene. Charles Weizmann.

A low-molecular-weight olefin is passed to a finely divided metal hydrogenating catalyst, such as copper, at atmospheric pressure, a temperature between 600-725° C. and at a space velocity of 0.01-0.1 (on a liquid basis). The resulting products are fractionated and a principal fraction boiling between 120-150° C. is obtained which consists mainly of styrene and ethylbenzene.

U.S.P. 2,385,484. Process for the Production of Diolefins. K. A. Wright to Shell Development Company.

Diolefins such as butadiene, are produced from the corresponding mono-olefins by contacting the latter in the presence of at least 2 mols of steam at a temperature above 580° C. and at a gaseous hourly space velocity between 300 and 3000 with a catalyst composed of a dehydrogenating oxide of iron promoted with rubidium in an amount equivalent to 0.9 percent and 5.5 percent calculated as the oxide based on the dehydrogenating metal oxide.

U.S.P. 2,385,555. Cyclohexene Production. H. H. Voge to Shell Development Company.

Cyclohexane is contacted with a metal halide of the Friedel-Crafts type under naphthene isomerizing conditions, converting it to methylcyclopentane. This product is contacted with a dehydrogenation catalyst under dehydrogenating conditions, for instance with a chromic oxide-aluminum oxide catalyst at about 500° C. and under atmospheric or increased pressure for 0.2 to 5 seconds, converting the methylcyclopentane to methylcyclopentenes. The latter are contacted with a solid olefin isomerizing catalyst under suitable conditions, for instance with activated alumina, bauxite or the like at 350-500° C. and at ordinary pressure, whereby the methylcyclopentenes are converted to cyclohexene.

U.S.P. 2,386,135. Production of Dienes. R. C. Morris and R. J. Moore to Shell Development Company.

Dienes are produced from normal mono-olefins with 6-12 C atoms by first isomerizing the mono-olefins to produce branched chain olefins, then non-catalytically cracking the product in the vapor phase at 650-850° C. and at a pressure below 150 psi for a time sufficient to gasify 15-18 percent thereof. C_4 and C_5 dienes are separated from the cracked product. Further a fraction of substantially the same boiling range as the isomerized product is separated from the cracked product. Aromatics and dienes are removed from this fraction and the resultant diene-free and aromatic-free fraction is recycled to the cracking step.

U.S.P. 2,386,310. Butadiene Production. K. H. Hachmuth to Phillips Petroleum Company.

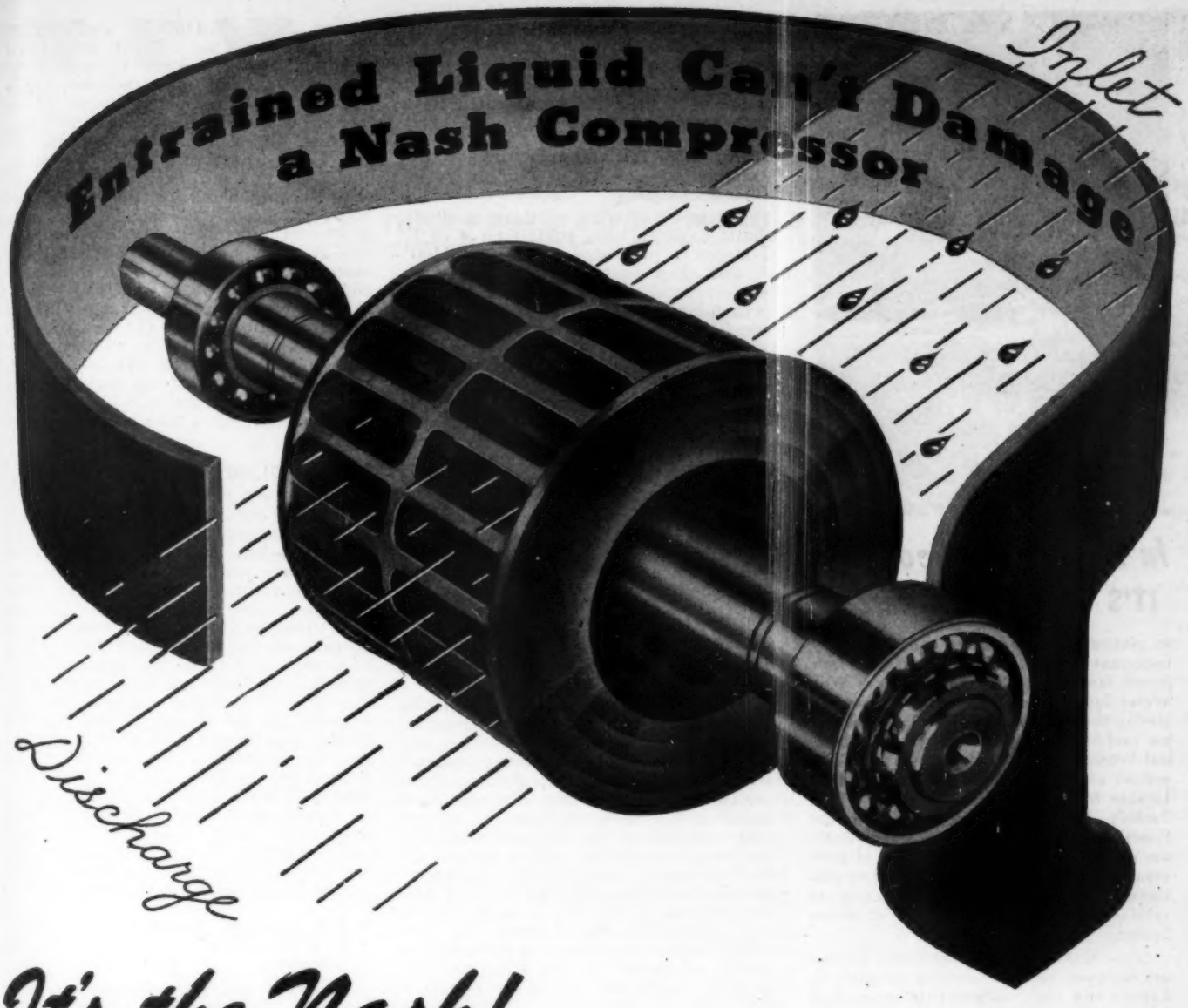
N-butane is first catalytically dehydrogenated to n-butene. The C_4 hydrocarbon content of the effluent obtained in this first-stage dehydrogenation is subjected to fractional distillation separating the butene-1 as an overhead product from a bottoms product containing n-butane and butene-2. The latter mixture is subjected to extractive distillation with a solvent which selectively dissolves the butene-2. The butene-2 is then stripped from its solvent. The overhead from the extractive distillation which is principally composed of n-butane is recycled to the first-stage dehydrogenation. The butene-1 is combined with the butene-2 and the mixture is subjected to a second-stage catalytic dehydrogenation, forming butadiene. Furfural is the preferred selective solvent used.

U.S.P. 2,386,947. Catalytic Dehydrogenation Process. K. H. Hachmuth to Phillips Petroleum Company.

Paraffins are dehydrogenated to olefins by contacting them with a suitable catalyst under dehydrogenating conditions. This treatment is initiated at a high temperature, e.g. 1160° F. which is gradually lowered from the beginning to the end of the on-stream period as the dehydrogenation proceeds and the catalyst becomes deactivated, e.g. at a rate of 1° F. per minute.

U.S.P. 2,387,524. Dehydrogenation Process. R. N. Meinert to Standard Oil Development Company.

Hydrocarbons such as butene are dehydrogenated continuously by preheating them to a temperature below hydrogenation temperature and then contacting them with steam (in an amount sufficient to reduce the partial pressure of the hydrocarbons to about 0.25 to 0.1 atmosphere) at 1200-1500° F. and in the presence of a catalyst for a short time (0.1-1 second). The catalyst consists of



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a major proportion of MgO and a minor proportion of Fe_2O_3, CuO and K_2O. Two catalyst chambers are provided, one being on stream while the catalyst in the other is being regenerated by a large excess of steam.

U.S.P. 2,387,731. Manufacture of Diolefins. J. G. Allen to Phillips Petroleum Company.

A gaseous mixture of steam and aliphatic hydrocarbons containing 4 to 12 C atoms is preheated to at least 1100° F. and then passed to a partial-combustion-reaction zone together with an oxygen-containing gas. The latter is first passed through an incandescent porous refractory material of large surface area impregnated with a metal oxide combustion catalyst. The preheated steam-hydrocarbon mixture is introduced into the reaction zone at a point immediately adjacent the catalyst coated surface and in a manner such that the products pass directly away from this surface to and through a reaction zone which is in free communication with but extends away from the said surface. The resulting low-boiling diolefin hydrocarbon mixture is cooled by quenching.

U.S.P. 2,388,078. Process for Conversion of N-butane to Butadiene. E. D. Reeves to Standard Oil Development Company.

N-butane is converted to butadiene by contacting it with a dehydrogenation catalyst under suitable conditions and then contacting the effluent mixture with a solvent to absorb hydrocarbons with at least 4 C atoms. The unreacted paraffins are removed from the solvent and recycled to the catalyst zone. The olefins are contacted with a second dehydrogenation catalyst. The hydrocarbon material thus obtained is mixed with that treated by the first catalyst prior to contacting with the solvent. The first dehydrogenation catalyst is composed of oxides of metals of the 6th group deposited on alumina and is maintained at 950-1150° F. The second dehydrogenation catalyst is maintained at 1050-1300° F. Methyl amine is the preferred solvent for the hydrocarbons.

U.S.P. 2,389,215. Process for Dehydrogenating a Hydrocarbon. H. M. Singleton to Standard Oil Development Company.

In the conversion of a hydrocarbon by dehydrogenation an organic heterocyclic compound containing a plurality of double bonds in its ring structure is added to the charge hydrocarbon. The mixture is subjected to the action of a dehydrogenating catalyst at 600-1400° F. The unsaturated heterocyclic compound (such as furan, thiophene, pyrrole and their derivatives) is acting as hydrogen acceptor.

U.S.P. 2,389,342. Catalytic Dehydrogenation. M. W. Conn to Phillips Petroleum Company.

A normally liquid condensate obtained from the hydrocarbon mixture occurring naturally in the gaseous phase in distillate wells under a pressure of more than 1000 psi is fractionated so that a first fraction containing the high-octane components in the motor-fuel boiling range is separated from a second fraction containing the low-octane and heavier components. This second fraction is passed in the presence of recycled hydrogen and C_3 and C_4 hydrocarbons in contact with a granular bauxite catalyst at about 900-1100° F. and 10-1500 psi pressure. The effluent from this treatment is fractionated to recover at least the following six fractions. (1) a fraction consisting of hydrogen (2) a fraction containing C_3 and lighter hydrocarbons, (3) a fraction containing C_3 and C_4 hydrocarbons, (4) a normally liquid fraction containing the high-octane components in the motor-fuel boiling range, (5) a second liquid fraction containing the low-octane components and components heavier than those named before, and (6) a fraction containing tars and heavy material unsuitable for recycling. At least a portion of the hydrogen fraction (1) and of fraction (3) is recycled to the catalytic conversion step. Fraction (5) is also recycled together with fresh material. The first high-octane fraction is blended with fraction (4) to form a high octane motor fuel.

U.S.P. 2,391,160. Refining Operation. C. A. Hillman and D. L. Campbell to Standard Oil Development Company.

Butadiene is produced continuously by heating n-butane to dehydrogenation temperature and then passing it in contact with a chromia-alumina catalyst for 0.05 to 2 seconds at 1000-1400° F. and at a pressure averaging 95 mm. Hg. A portion of the butane and butene-2 is then separated from the reaction products by fractionation and recycled to the dehydrogenation zone. The remainder of the C_4 cut of the reaction products is treated with methylamine to remove butadiene. The remainder is treated with sulfuric acid to extract isobutene. The residual mixture is alkylated by means of an alkylation catalyst such as sulfuric acid, converting isobutane and butenes to alkylate. After separating the latter, the remaining n-butane is recycled to the dehydrogenation zone.

U.S.P. 2,391,188. Butadiene Production. R. J. Patterson to Phillips Petroleum Company.

N-butane and/or n-butenes are catalytically dehydrogenated to form butadiene. After removing the butadiene formed from the reaction mixture, a fraction consisting of isobutylene and butene-1 is separated from butene-2. The butene-2 is recycled to the dehydrogenation step. The isobutylene and butene-1 fraction is subjected to isomerization. Butene-2 in the isomerization product is separated from the isobutylene by fractional distillation. The residual isobutylene, which is now substantially free from butene-1 is treated in such a manner as to partially isomerize it to n-butene comprising a major portion of butene-2. The isomerization product is separated by fractional distillation into a fraction containing the unconverted isobutylene and a fraction of butene-2. The isobutylene fraction is recycled to the isomerization step, while the butene-2 fraction is recycled to the dehydrogenation step.

U.S.P. 2,391,646. Process for Dehydrogenating Olefins. W. A. Schulze, J. C. Hillyer and H. E. Drennan to Phillips Petroleum Company.

Aliphatic diolefins, e.g. butadiene, are obtained from corresponding monoolefins by contacting the latter at 1100-1300° F. with bauxite impregnated with minor proportions of MgO and of $Ba(OH)_2$ or $Sr(OH)_2$, e.g. with 1-10 percent of each of the active ingredients. A pressure of 0-200 psi is employed in the dehydrogenation step.

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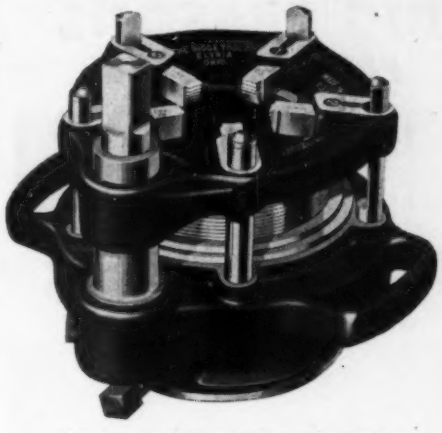
1—Pipe Threader

A quick-setting workholder and new balanced loop handles are two innovations on the new 4P geared pipe threader announced by The Ridge Tool Company, Elyria, Ohio. The quick-setting workholder affords an advantage in enabling a worker to set the threader exactly to size before being placed on the pipe; once on the pipe only one screw needs to be tightened to hold it securely. All bothersome bushings have been eliminated. The balanced loop handles on the enclosed gear case prevent slipping or tipping over, making it easy to pick up and put straight on the pipe, and easy to carry with one hand.

The manufacturer also claims economy of operation by virtue of a twin-anchored drive shaft that turns in oilless bronze bearings. The gear is fully enclosed, packed in grease, protected from dirt and wear.

Precision made of rugged steel-and-malleable, the new 4P is provided with four sets of five high-speed steel dies to cut clean, accurate threads, standard or tapered, drip threads and short nipples. A ratchet handle is provided with each 4P and a special "ridged" design universal drive shaft is available for power drive. Production is said to be still limited, distribution through supply houses only.

CHECK THE CARD AT PAGE 249



Ridge Tool 4P Geared Pipe Threader

2—Controller

The Foxboro Company, Foxboro, Massachusetts, has announced its Model 40 controller, which it says represents no departure in basic operating principles from those of Model 30, introduced in 1942, but embodying improvements in design and construction which offer new advantages important and numerous enough to characterize a wholly new instrument.

Features of the Model 40 may be generalized, the announcement says, as a simplification of design, with more sturdy unit construction, resulting in superior performance, easier and more flexible adjustments, and new convenience in servicing, including easy replacement or interchange of unit parts.

The operating-panel feature of "Stabilog" control instruments is retained, and when the case door is open every part normally requiring attention is immediately accessible without removing the chart.

An outstanding characteristic of the Model 40 is its adaptability in changing from one type of control to another. Setting of the proportional band, from 0 to 200 percent or higher, is easily made by the turn of a thumb wheel. Reversal of instrument action is made in a like manner. The range of reset is 500:1 and, although not considered critical, is a continuous adjustment. Elimination of the reset and derivative functions is simple. A much-improved adjusting device permits zeroing the pen without realigning the control point. A new-type transfer switch permits change from manual to automatic control, or vice versa, with no valve motion.

The die-cast aluminum case, similar to Model 30, fits the same opening in an instrument panel. Hasp and hinges of new design facilitate door opening or removal, and also insure tight closure.

Improvements in design have been carried out in all the associated equipment, as well as in the primary "Stabilog" control mechanism, including the "Pneumaticset" and the ratio-control assemblies and the units for pneumatic transmission, it was said.

Model 40 is made as an indicator as well as a single-pen and multi-pen recorder, and is available for on-off control action, proportional, proportional and reset ("Stabilog"), proportional with derivative, and proportional and reset with derivative ("Hyper-Reset").

All types are described, and numerous illustrations reveal their constructional features, in Bulletin 381.

CHECK THE CARD AT PAGE 249

Foxboro Model 40 Controller

3—Diaphragm Regulating Valve

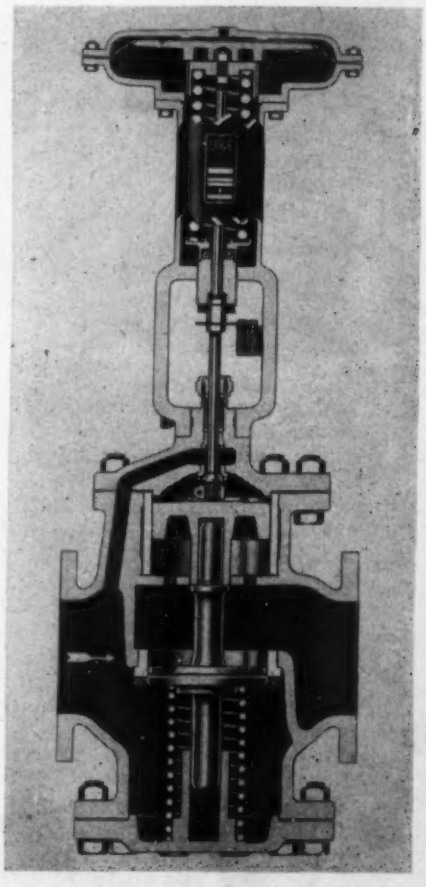
Leslie Company, 169 Delafield Avenue, Lyndhurst, New Jersey, has announced a new line of single-seated, fully balanced regulating valves.

These diaphragm valves are made with cast-iron, bronze, and steel bodies for steam, air or gas pressures up to 1000 psi, and are designed to be actuated by standard pressure or temperature instruments or controllers using 20 psi air or clean water.

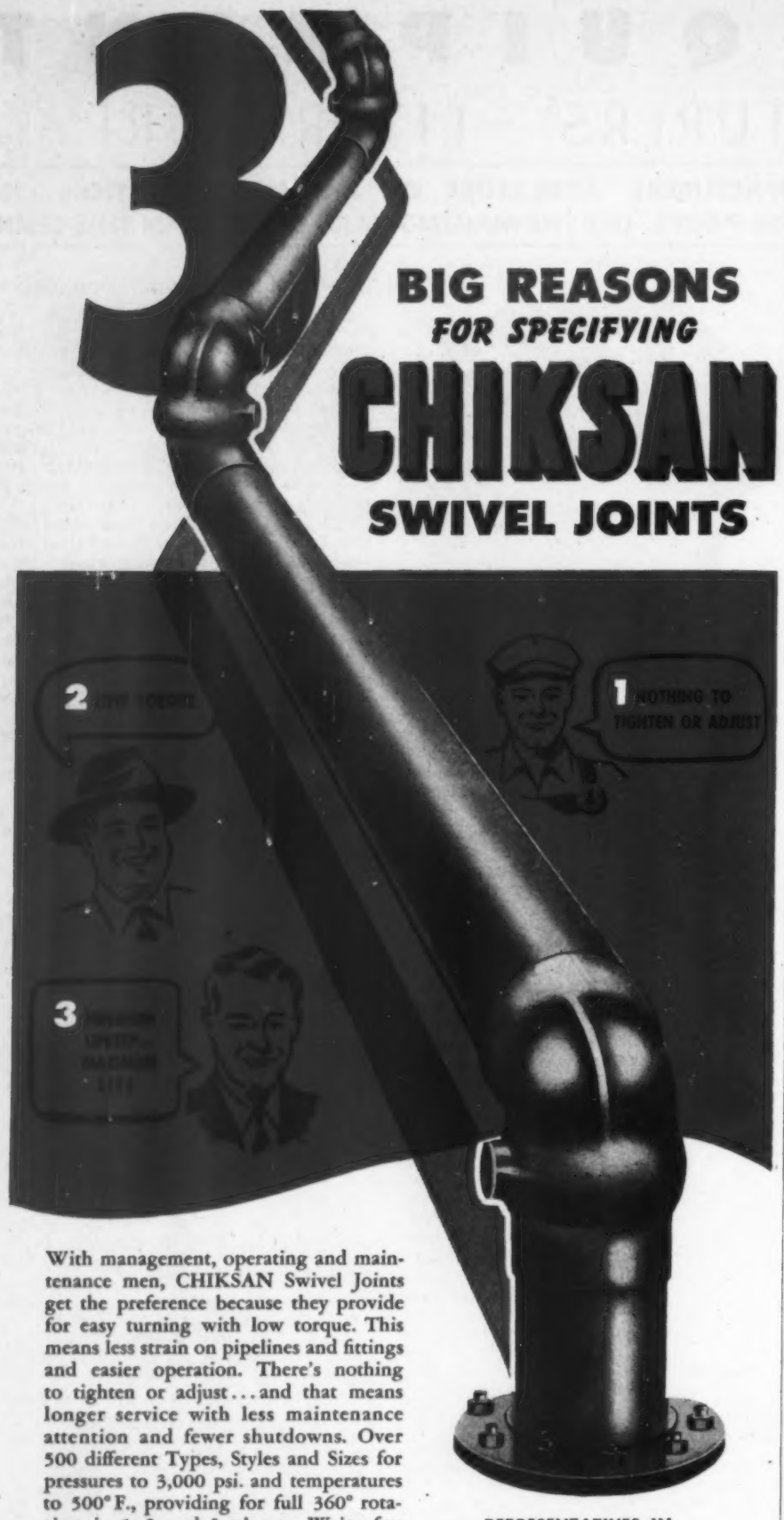
Supplied as standard equipment with 800 Brinell hardened stainless steel main valves and stellited seat rings, these single-seated valves provide equal or greater capacities than most double-seated regulating valves of the same size, it is claimed, and have the additional advantage of being tight in dead-end service.

For temperatures over 450° F., steel diaphragm valves are equipped with a newly designed cooling bonnet that offers a large radiating surface and a reservoir of condensate to aid in keeping the stuffing-box temperature down.

These diaphragm valves, designated as Classes D-1 (cast iron or bronze) and DS-1 (steel) are normally supplied with the valve below the seat in closed posi-



Leslie Class D-1 Diaphragm Regulating Valve



With management, operating and maintenance men, CHIKSAN Swivel Joints get the preference because they provide for easy turning with low torque. This means less strain on pipelines and fittings and easier operation. There's nothing to tighten or adjust... and that means longer service with less maintenance attention and fewer shutdowns. Over 500 different Types, Styles and Sizes for pressures to 3,000 psi. and temperatures to 500°F., providing for full 360° rotation in 1, 2 and 3 planes. Write for latest catalog.

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BALL BEARING SWIVEL JOINTS FOR ALL PURPOSES
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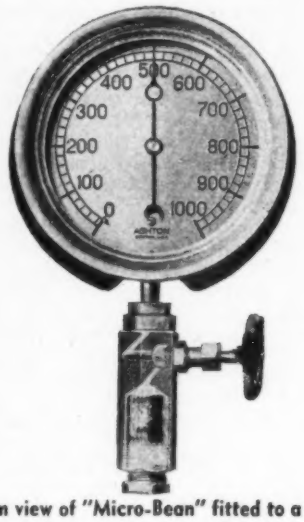
New York 7 Houston 2

tion, opening with diaphragm pressure. However, by use of a new type reversing superstructure, the valve can be changed to a normally open below the seat position, closing with diaphragm pressure, without changing internal parts, removing main body from pipe line, or cutting valve stems.

CHECK THE CARD AT PAGE 249

4—Pulsation Dampener

J. A. Campbell Company, 645 East Wardlow Road, Long Beach 7, California, has announced availability of an improved pulsation dampener for gauges and governors. The "Micro-Bean" is available in all-brass and all-steel with stainless-steel stem. No piston is required. Temperature has no effect on the viscosity and surface tension of liquids within the line. The manufacturer states that in all pulsation dampeners scoring is inevitable because of the extreme viscosity through the restricted passage, and that most dampeners have a tendency to score out after a year or two. To overcome this problem, the "Micro-Bean" is provided with a screw which when turned slightly will bring back the seating to perfect control. This can be repeated every two years or so. When the micro-plug eventually becomes scored up the scores can be eradicated by opening and closing the valve tightly about 10 times.



Phantom view of "Micro-Bean" fitted to a gauge

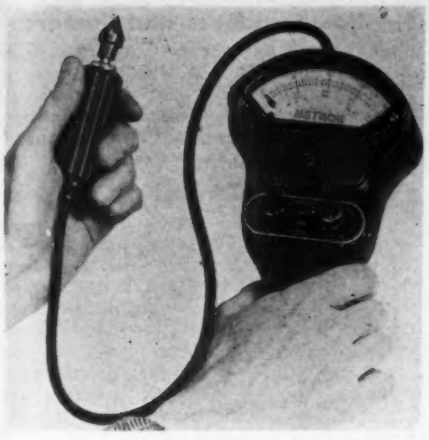
The "Micro-Bean" is stocked in ¼-, ⅜- and ½-inch, for pressures up to 6000 pounds.

CHECK THE CARD AT PAGE 249

5—Low-Speed Tachometer

Metron Instrument Company, 432 Lincoln Street, Denver 9, Colorado, has announced availability of a new low-speed hand tachometer, Type 25B, for direct measurement of speeds down to 10 revolutions per minute and 1 foot per minute. It has three ranges of 10 to 200, 20 to 400, and 50 to 1000 revolutions per minute.

This tachometer consists of the head, which is normally held in the left hand, and the indicating unit which is normally held in the right hand when making measurements. This arrangement is convenient when measuring speeds of rotating shafts in inaccessible places. Finger-tip control is provided on the indicating unit so that the speed range can be easily changed by the thumb of the



Metron Low-Speed Tachometer

right hand while making a measurement. No damage will result to the instrument if the speed range is varied while the tachometer head is engaged with the rotating shaft. Also, the tachometer cannot be damaged by overspeeding.

The tachometer head is a contacting-making mechanism similar to the blades of a relay. It is, in effect, a double-pole, double-throw switch which is operated by the rotation of the shaft or spindle extending from the tachometer head. When this spindle rotates, the switch blades oscillate, and thus the direction of rotation of the shaft does not affect the reading. This double-pole, double-throw switch is connected in a series circuit consisting of a battery, a condensor and a milliammeter. Oscillation of the switch blades alternately charges and discharges the condensor by means of the battery. This permits current to flow in small charges from the battery through the milliammeter. The circuit is designed so that the current is exactly proportional to the speed of the spindle. The circuit values are chosen so that the accuracy is not dependent on the time of contact dwell or on the contact resistance. The meter does not respond to each charge but reads average current instead.

The accuracy of this tachometer is 1 percent of full scale and is ruggedly built for sustained accuracy. The head contains only one rotating part which is mounted in permanently lubricated ball bearings. This instrument is supplied complete in a carrying case with contact tips and discs for measuring linear speeds.

Low- and high-speed adapters are available for this tachometer for extending its range down to 1 and up to 10,000 revolutions per minute.

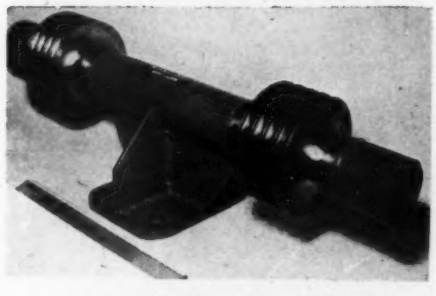
CHECK THE CARD AT PAGE 249

6—Expansion Joint

MagniLastic Division of Cook Electric Company, 2700 North Southport Avenue, Chicago 14, Ill., announces its new anchor joint, latest in its line of packless expansion joints.

The anchor joint combines rigid anchoring of piping at accessible points, with allowance for unequal thermal expansion and contraction on either side of the anchor point, due to differences in pipe length, ambient temperatures, exposure, etc. This is accomplished by varying the number of bellows flanges on either side of the anchor base to accommodate each condition encountered. Where severe hydraulic surges within piping occur, anchor joints also minimize the transmission of shock to fittings and adjacent structures.

MagniLastic Anchor Joints are made for standard pipe sizes from ½ to 24-inch. The expansion flanges are supplied from any one of the standard MagniLastic 25-, 55-, 150-, 400- or 1000-pound series, to accommodate all pressures from vacuum to 1000 psi. Joints of stainless steel, Monel, Inconel and copper cover all commonly-met corrosive conditions. Temperatures handled



MagniLastic Anchor Joint

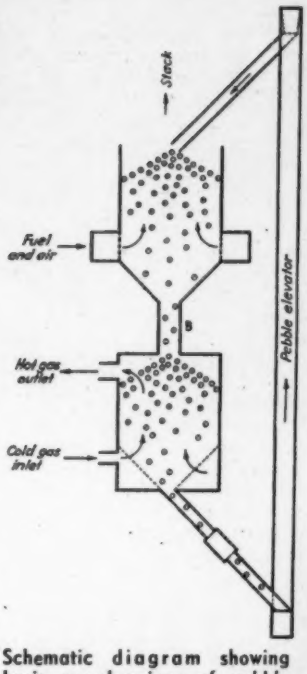
ranged from —300° F. to 1600° F. A variety of standard fittings such as elbows, tees, ells, Y's, etc., can be incorporated into anchor joints to make a single, compact, accessible unit. Welding ends are regularly supplied, with pipe flanges and inner linings optional.

Anchor joints are applicable to all forms of indoor and outdoor piping systems. Standard anchor base dimensions for all pipe sizes, and quotations for specific requirements, may be obtained by writing the manufacturer.

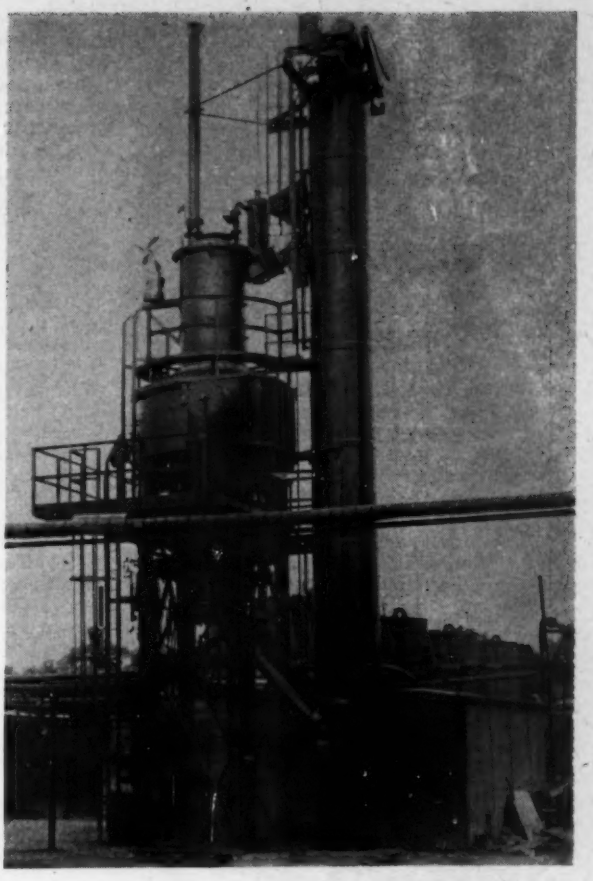
CHECK THE CARD AT PAGE 249

7—Chemical Feeder

The American Water Softener Company, Lehigh Avenue and Fourth Street, Philadelphia, has available an illustrated bulletin on Corrective Chemical Feeding.



Schematic diagram showing basic mechanism of pebble heater developed by The Babcock & Wilcox Company, as illustrated in the photograph.



8—Heat-Transfer Unit

The Babcock & Wilcox Company, 85 Liberty Street, New York 6, has developed a new form of heat-transfer unit, called the "pebble heater," capable of heating gases above the operating temperatures permissible in metallic heat interchangers. The principle involved consists of raising to high temperatures a constantly moving column of pebbles made of a heat-resistant non-metallic material, and then passing the gas to be heated through the interstices of the moving pebble column. The unit has been used to heat air to 2300° F. and steam to 1800° F., it was said.

The heater is described as consisting of two cylindrical chambers, one above the other, connected by a throat section of reduced diameter. By filling the chamber and throat with refractory pebbles, permeable beds are formed through which gases can be made to travel. At the outlet of the bottom chamber, there is a mechanical feeder which controls the rate at which the pebbles are conveyed continuously back to the top of the upper chamber. The pebbles in the heater move downward at a constant rate during the entire operation.

Fuel and air are burned in a combustion chamber, and the products of combustion flow through hot gas inlets into the top chamber and then upward through the bed of downwardly moving pellets and out the stack at the top. The heated pebbles pass through the throat section to the lower chamber. At a point near the bottom of the lower chamber, the gas to be heated is introduced. The gases pass through a screen to provide uniform distribution, then travel upwardly through the heated pebble bed and leave at a point near the top of the lower chamber.

In operation, the pressures in the two chambers usually are maintained at

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the same value, so that there is no gas flow through the throat section. Zero gas flow, or a flow in either direction, can be maintained by automatic control of a damper in the exit stack of the upper chamber.

"One of the first applications of the pebble heater," an announcement said, "has been for heating the air used for combustion of fuels. By using highly preheated air, temperatures can be reached which generally have been considered to belong in the field of electric furnaces, and far exceed temperatures required for melting steel.

"In the field of refractories, which covers the making of materials for furnace constructions, a continuous melting operation has been developed in which furnace construction materials can be melted and poured like molten steel, to give them even greater temperature resistance. Another interesting development in connection with the melting of refractories is the production of a refractory mineral fibre which is capable of a very high temperature use limit. Mineral fibres made with this apparatus can be used in chemically corrosive applications, for filtering out dust from high-temperature gas streams, and as a packing material to fill expansion joints in refractory furnace construction. These fibres can also be used to advantage as heat insulation where weight is important, such as the insulation of jet motors.

"In the general field of process chemistry, the pebble heater, because of its large heat transfer surface, is particularly suited for rapid heating of gases to high temperatures."

CHECK THE CARD AT PAGE 249

9—Gas Scrubbers and Coolers

Peabody Engineering Corporation, 580 Fifth Avenue, New York 19, has issued Bulletin No. H-202, on Peabody gas scrubbers and coolers. It summarizes recent developments and applications.

CHECK THE CARD AT PAGE 249

10—Process Controllers

Bailey Meter Company, 1050 Ivanhoe Road, Cleveland 10, Ohio, has issued its Bulletin 17, in which process controllers, control components, measuring components and instrument combinations are briefly described and illustrated. Application data such as sizes, pressure standards and ranges are listed for the various components. Typical control systems of both single- and multiple-element types are illustrated.

CHECK THE CARD AT PAGE 249

11—Portable Pyrometers

Wheelco Instrument Company, 847 West Harrison Street, Chicago 7, has available Bulletin D602-4, which describes the company's line of portable pyrometers. Specifications for selection of extension holders, adapters and thermocouple tips are listed in chart form.

CHECK THE CARD AT PAGE 249

12—Gas-Diesel Engine

The Cooper-Bessemer Corporation, Mt. Vernon, Ohio, is distributing Bulletin No. L-51, which gives engineering specifications of its Type LS Gas-Diesel Engine.

CHECK THE CARD AT PAGE 249

13—Compress Units

The Cooper-Bessemer Corporation, Mt. Vernon, Ohio, is distributing Bulletin No. 338, descriptive of its Type GMX Compressor Unit which was recently announced. Photographs and colored sectional drawings are included with full engineering details.

CHECK THE CARD AT PAGE 249

14—Floor Treatments

Truscon Laboratories, Inc., Caniff and G. T. Railroad, Detroit 11, Michigan, has issued a new specification book covering floor treatments — concrete and wood.

CHECK THE CARD AT PAGE 249

15—Packing

Raybestos-Manhattan, Inc., Manheim, Pennsylvania, has issued a 64-page catalog which includes illustrated descriptions of its line of mechanical packings, together with charts showing types of service for which each style of packing is recommended.

CHECK THE CARD AT PAGE 249

16—Thermometers

Palmer Thermometers, Inc., 2501 Norwood Avenue, Cincinnati 12, Ohio, has issued Bulletins 46-2 and 46-3 which give detailed description and illustration of thermometers of the company's manufacture.

CHECK THE CARD AT PAGE 249

17—Turbo-Blowers, Compressors

Allis-Chalmers Manufacturing Company, Milwaukee 1, Wisconsin, has issued a 16-page booklet designed for student training, which explains the workings of turbo-blowers, rotary compressors and vacuum pumps. The booklet shows construction of the company's turbo-blower and tells why it is important that complete information be furnished to figure blowers correctly. Reproduced are curves depicting pressure

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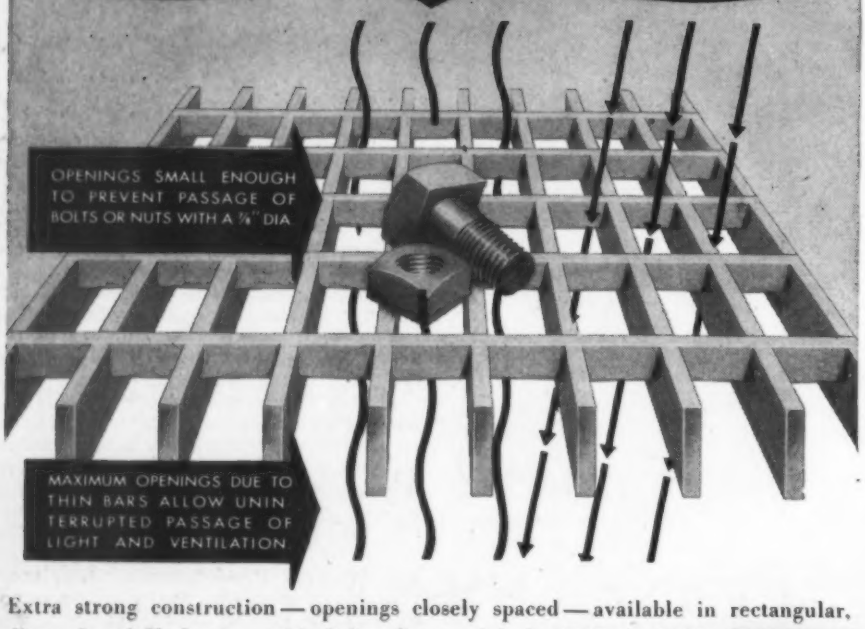
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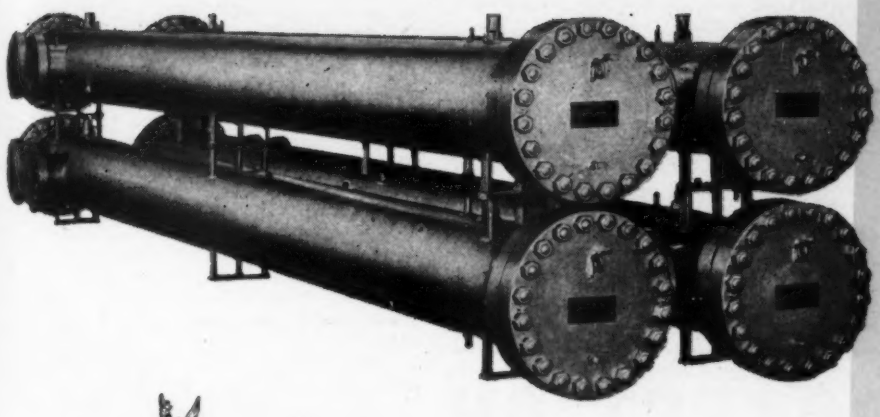

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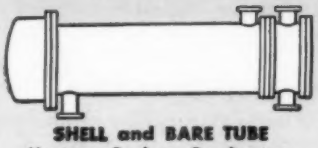

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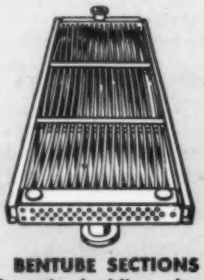

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volume, influence of water vapor on volume, and correction curves for volumes to maintain constant air weight with changes in temperature and barometric pressure.

CHECK THE CARD AT PAGE 249

18—Resinous Cement

Nukem Products Corporation, Buffalo 20, New York, has available an informative folder descriptive of Nukem All-Purpose Resinous Cement. It includes a resistivity chart, and gives pertinent data on physical properties and application.

CHECK THE CARD AT PAGE 249

19—Laboratory Pilot Plant

Fansteel Metallurgical Corporation, North Chicago, Illinois, is distributing Form F-540, descriptive of its Tantalum Acid-Proof Laboratory Pilot Plant.

20—Gaskets

United States Gasket Company, Camden, New Jersey, has issued a new bulletin on its Ajax spiral wound metal and asbestos gaskets. This booklet contains a wealth of engineering data on construction and application on various types of gaskets.

CHECK THE CARD AT PAGE 249

21—Bus Duct

Westinghouse Electric Corporation, Box 868, Pittsburgh 30, has issued a 40-page booklet (B-3714) to aid in selection and application of a bus duct system to provide flexible, accessible power in industrial plants. It diagrams and analyzes step by step three typical bus duct installations. Drawings and photographs of the duct components illustrate installation, construction and design of the equipment. The engineering data section of the booklet includes standard ratings of main breakers, temperature conversion tables, formulas for determining amperes, horsepower, kilowatts and kilovolt-amperes, and load power factor curves, and other information designed to aid in correctly applying bus duct, and in selecting power take-off devices.

CHECK THE CARD AT PAGE 249

22—Expansion Joints

MagniLastic Division of Cook Electric Company, 2700 Southport Avenue, Chicago 14, Illinois, has revised and enlarged its Catalog 265M, which lists complete engineering data, dimensional tables and specifications for MagniLastic's standard packless expansion joints, anchor joints and low-pressure large-diameter expansion joints. It contains information on other recent development of the company's products, such as offset, turbo and jacket joints, and includes material on high-temperature exhaust bellows and heavy-duty clinch-lock bellows.

CHECK THE CARD AT PAGE 249

23—Squirrel-Cage Induction Motors

The Louis Allis Company, Milwaukee 7, Wisconsin, has released a new Bulletin No. 720, covering its Type OG (protected type) standard squirrel-cage induction motor. This bulletin, handsomely illustrated with sectional photographs, fully covers construction, features, and some typical applications of the motor.

CHECK THE CARD AT PAGE 249

24—Magnesia Insulation

The Magnesia Insulation Manufacturers Association, 1317 F Street, N. W., Washington 4, D. C., recently has issued No. 1 of NIMA News, to be issued quarterly. Announced purpose is to establish a helpful medium of contact between users of 85% magnesia insulation and the manufacturers, and to publish helpful information about heat insulation.

CHECK THE CARD AT PAGE 249

25—Refractories

Chas. Taylor Sons Company, Cincinnati, Ohio, has available an informative illustrated booklet, Bulletin 201, on Taylor Zircon refractories for high-temperature service and special applications. This bulletin should interest anyone concerned with the specification or use of refractory materials.

CHECK THE CARD AT PAGE 249

26—Vibration Insulators

The B. F. Goodrich Company, Akron, Ohio, has available a new bulletin on its line of "Vibro-Insulators," devices of rubber and metal to cushion equipment and reduce vibration and noise.

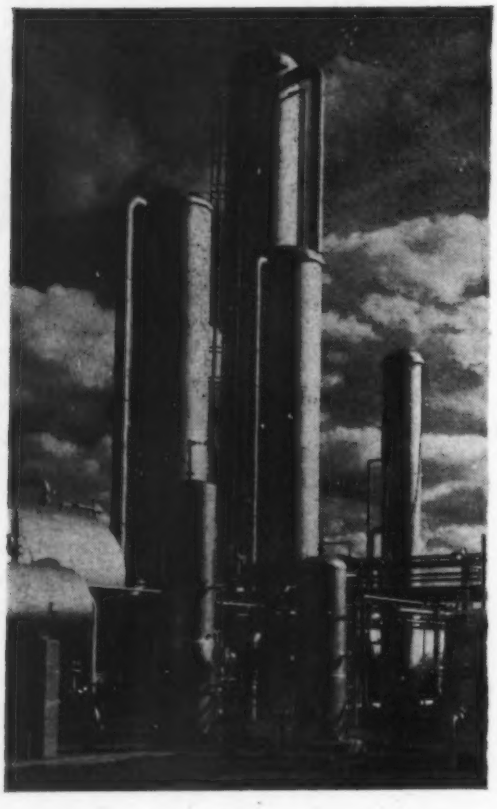
CHECK THE CARD AT PAGE 249

27—Alloy Metal Wire

Alloy Metal Wire Company, Prospect Park, Pennsylvania, has issued a new catalog, D-2, on wire, rod and strip. It includes engineering information on strength, electrical resistivity, modulus of elasticity, heat treatment, temperature limits, and magnetic properties. The metals covered are the high-nickel-content alloys, Monel, nickel, "Z" nickel, "L" nickel, Inconel, and specialized Monels ("K," "R" and "KR") for machining and heat-treating properties.

CHECK THE CARD AT PAGE 249

YOU BUY PROTECTION



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- Obviates need for steam in header boxes
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- Permits faster "heading-up"
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About EQUIPMENT—SERVICE Suppliers

International Nickel Opens Texas Technical Section

The International Nickel Company, Inc., has opened a Texas technical section of its development and research division, in the Bankers Mortgage Building, Houston, with R. J. Rice, metallurgical and chemical engineer, in charge. The section will furnish industry technical information and assistance relating to alloys containing nickel. Its activities will cover Texas, Oklahoma, Louisiana and the south half of Arkansas.

R. J. Rice

Rice was associated with Beaumont Iron Works Company from 1933 to 1938, responsible for operation of that company's metallurgical department, melting shop, heat-treating department and specifications for steels and irons. Later he was associated with Metal Goods Corporation, Houston, as foundry engineer and metallurgist, directing his activities toward use of nickel in iron and steel foundries and industrial application of nickel-containing alloys. In January, 1942, he went to Washington as senior industrial specialist in the manganese-chrome branch of WPB, and the following October was commissioned lieutenant (j.g.) and assigned to the Navy's office of procurement and material, with additional duties on the staff of the Army-Navy munitions board. In October, 1945, he was assigned as seizure officer at The Standard Oil Company's Toledo, Ohio, refinery, and later in the same capacity at Phillips Petroleum Company's Kansas City, Kansas, plant. He was released from the Navy early this year with the rank of lieutenant commander.

McConnor Succeeds Goble as National Tube Sales Head

William F. McConnor has been named vice president in charge of sales of National Tube Company, succeeding John E. Goble, who has been elected president of the company. Goble succeeds Charles R. Cox, who moved to the presidency of Carnegie-Illinois Steel Corporation, the latter position having been made vacant when J. Lester Perry became assistant to the president of United States Steel Corporation, of which the two mentioned companies are subsidiary.

J. E. Goble

W. F. McConnor

McConnor began his career as an apprentice engineer with York Manufacturing Company, in 1914, following graduation from Lehigh University. He went with National Transit Company in 1915, as a draftsman and designer on engines for pipe lines. Two years later he joined National Tube Company's Pittsburgh engineering department. In 1920 he was made assistant superintendent of the large pipe department, and from 1923 to 1931 he was an assistant to the vice president on steel distribution and seamless pipe. He was assistant general manager of sales from 1931 to 1936, when he became general manager of sales.

Goble has been connected with United States Steel Corporation subsidiaries 21 years, having joined Oil Well Supply Company in 1925 as South American representative working out of Maracaibo. In 1929 he became assistant manager of export sales at New York, two years later became manager of the export department, and remained in that position until elected vice president of National Tube Company in 1936.

Slater Manager of Tank Sales For American Car and Foundry

R. S. Slater has been appointed manager of tank-car sales for American Car and Foundry Company, and as such will be in charge of sales of tank cars, storage tanks and pressure vessels. He will continue his headquarters at New York.

Slater started his career with American Car and Foundry Company in 1923 as an expediter in the material assembly division of the purchasing department in New York. In 1935 he transferred to the sales department and in 1936 was appointed sales agent in the New York sales department.

Rockwell Manufacturing Company Adds Hydraulic Division

A new Hydraulics Division has been established by Rockwell Manufacturing Company, Pittsburgh. W. H. Marsh has been appointed general manager of the division, which will specialize in the development of hydraulic equipment.

W. H. Marsh

Marsh attended Cornell University where he studied mechanical engineering. He joined Pittsburgh Equitable Meter Division of Rockwell Manufacturing Company in 1934 after previous experience with

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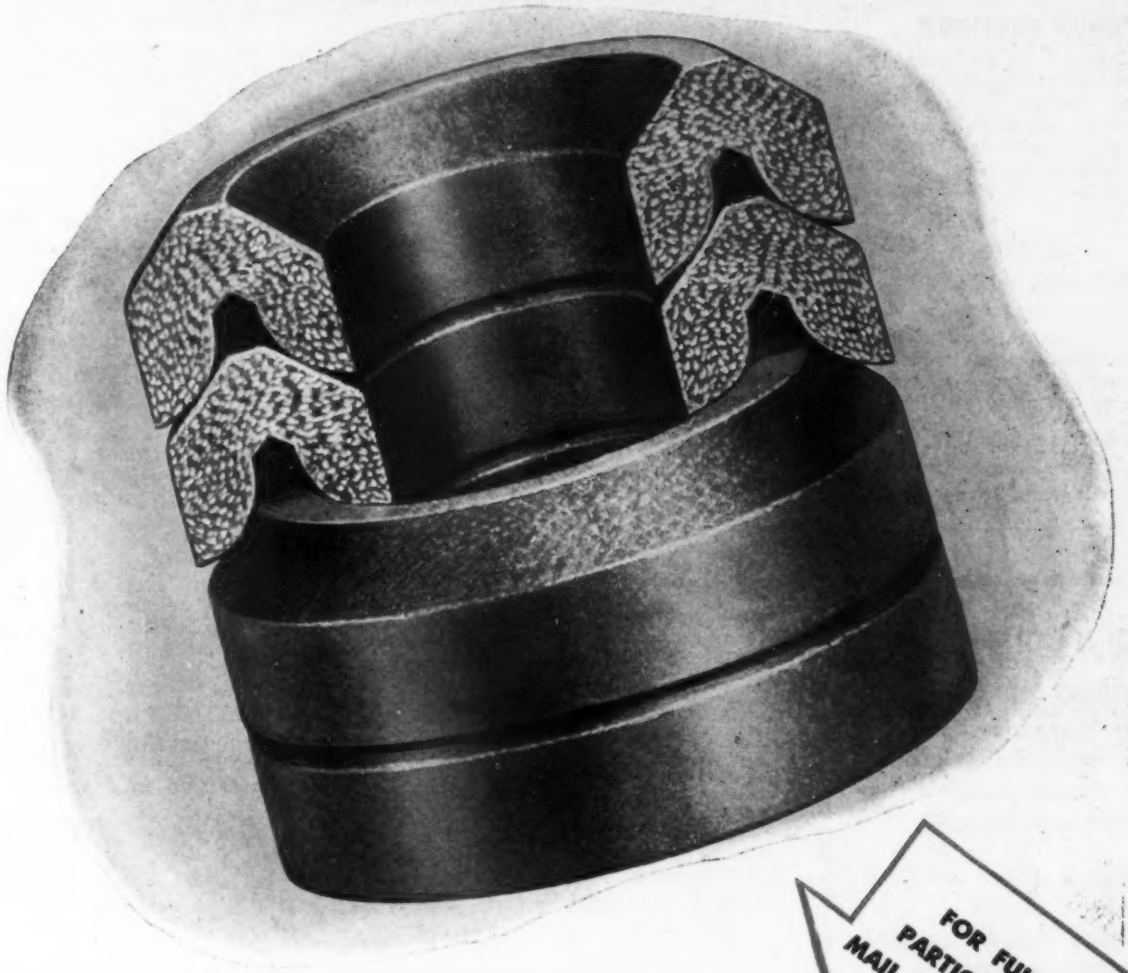

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The Hydraulics Division, it was explained, is an outgrowth of the gun-pointing program Marsh directed during the war, and which was closely aligned with a standard product of Rockwell Manufacturing Company.

E. R. Gilmore, who has been chief engineer of Emco products, has been appointed chief engineer of the Pittsburgh Equitable Meter Division. He was graduated from Rensselaer Polytechnic Institute in 1920 and since that time has been engaged in the field of measurement and control.

Ensminger Head Mathieson Fire Protection Department

The Mathieson Alkali Works has entered the fire-protection field with both high- and low-pressure carbon dioxide equipment. The company operates a large carbon dioxide plant at Saltville, Virginia.

Harry Ensminger

C. T. Longaker, manager of the company's carbon dioxide division, has named Harry Ensminger to head the fire-protection department, with headquarters at the New York office, 60 East 42nd Street.

The company proposes to specialize in built-in systems, and its engineering service will include surveys to determine fire-protection requirements, plans for systems, and supervision of installation. For large users, recharging problems will be simplified by the company's recently announced dry-ice liquefier which has a capacity of 1000 pounds of carbon dioxide. For others, recharging facilities are available at 16 distribution centers.

Ensminger became affiliated with The Mathieson Alkali Works in 1945. Prior to that time he was sales manager of the Electronics Division of Federal Telephone and Radio, and before that was sales manager for Cordox Corporation for about six years.

Karl Johnson Moves to Chicago With J. F. Pritchard Company

Karl Johnson, sales engineer representing all divisions of J. F. Pritchard & Company, recently has been transferred to the company's Chicago office. He has been with the company's Tulsa office since January 1, shortly after his discharge from Navy service.

Karl Johnson

Johnson received his degree in mechanical engineering from University of Kansas in 1938. His experience includes a year with a consulting engineering company in Kansas City, and two years with Westinghouse Electric Corporation before entering the Navy. During four years of the latter service he was an engineering officer in both the Atlantic and Pacific theaters. He was put on inactive status as a lieutenant (senior grade).

Scamman Joins Houston Oil Field Material Company Staff

Charles W. Scamman has joined Houston Oil Field Material Company, Houston, and is connected with its industrial sales department which specializes in heavy chemicals.

C. W. Scamman

Scamman graduated from The Rice Institute in 1925 and for a number of years was associated with Stanley Gill, consulting engineer at Houston. For the past four years he has been associated with J. S. Abercrombie Company and Harrison Oil Company as engineer on construction of their plant at Sweeney, Texas. While on that job he served as engineer and advisor for the purchasing department on engineering products.

Beaumont Laboratory Offers Equipment Inspection Service

The Industrial Radiography Laboratory recently was opened in Beaumont for the purpose of serving the petroleum industry in the detection of corrosion and erosion actions in equipment through the use of electronic and other similar methods. J. H. Stewart, president, formerly was chief chemist and assistant metallurgist with Beaumont Iron Works Company.

J. H. Stewart

Stewart is a member of the Texas-Louisiana Gulf Section of the American Chemical Society.

Chavigny Opens Equipment Sales Office at Houston

Charles L. Chavigny, operating as C. L. Chavigny Company, has opened offices in the Sterling Building, Houston, to act as sales representative of manufacturers of specialized types of processing equipment for oil-refining and chemical plants.

Chavigny recently was placed on inactive duty with the rank of lieutenant commanded, after 3½ years in the Navy.

Johnson Joins A. O. Smith Corporation at Los Angeles

W. Stuart Johnson has joined A. O. Smith Corporation as manager of hydraulic products sales in the Pacific Coast district. He will make his headquarters in the Los Angeles office of the company.

W. S. Johnson

Johnson previously was with Southern California Edison, Inc., as engineer in charge of pumping equipment. He was a major in the war, serving as a production engineering offi-

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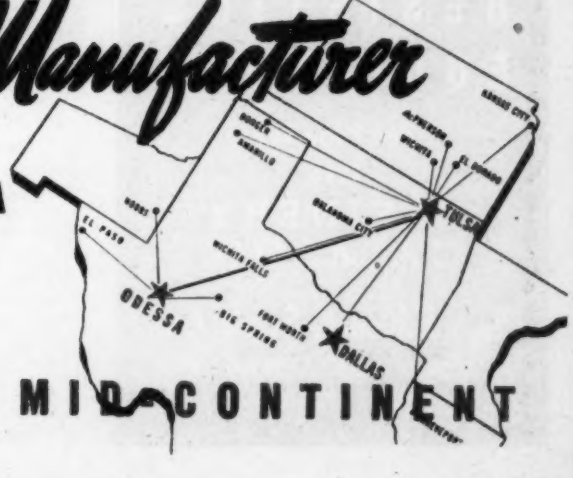

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cer with the air technical service command. Prior to his war service, he was associated with the Byron Jackson Company and Ingersoll-Rand Company.

He is a mechanical engineering graduate of California Institute of Technology at Pasadena, California.

Ray Miller Goes With Du Pont at Wilmington

Ray E. Miller, vice president of Warren Petroleum Corporation, in charge of Gulf Coast sales, has become associated with E. I. du Pont de Nemours & Company, and will have headquarters at Wilmington. Miller will be sales director of the chemicals division of the organic chemicals department. He will set up a new department to handle sales to the petroleum industry.

Miller was a vice president of Hanlon-Buchanan, Inc., Tulsa, at the time of its merger with the Warren organization. He had been in Houston only a few weeks prior to joining the du Pont organization on September 1. He has been active in natural-gasoline circles over a long period, having served as both secretary and president of the Natural Gasoline Association of America.

Farley, Schlegel Get New Posts With Brown Instrument Company

I. K. Farley has been named petroleum industry sales manager of The Brown Instrument Company, Philadelphia.

R. A. Schlegel has been transferred from Chicago to the New York office where he will assume duties formerly under supervision of Farley.

Farley will coordinate field sales activities in the petroleum industry and conduct market studies. He will make his headquarters at the Philadelphia main office of the company. Schlegel will have charge of petroleum accounts in the New York area.

Hovey Named Vice President The Harshaw Chemical Company

Waldo C. Hovey has been elected vice president in charge of research and development of The Harshaw Chemical Company, Cleveland, Ohio.

Hovey graduated with a degree in chemical engineering from McGill University in 1916. From 1916 to 1925 he was connected with Shawinigan Chemicals Corporation as superintendent; from 1925 to 1944 with Niacet Chemicals Corporation, Niagara Falls, New York, as general manager, and since 1944 has been manager of plant operations for Wyeth, Incorporated, Philadelphia.

Conaway Made Dallas Manager, New York Belt & Packing Company

J. E. Conaway has been named manager of the Dallas district office of New York Belting & Packing Company, succeeding Walter E. Belcher who has retired after 51 years with the company.

J. E. Conaway

Conaway has been assistant to Belcher working out of Memphis. As manager at Dallas he will supervise sales in Texas, Louisiana, Mississippi, Arkansas, Oklahoma, and parts of Kansas and Tennessee.

New York Belting & Packing Company also recently has appointed George G. Deverall as sales representative in New England, New York, New Jersey and Eastern Pennsylvania, with headquarters at the general offices in Passaic, New Jersey.

Deverall recently was released from military service after serving since 1940 in the Pacific. Since 1943 he was on General MacArthur's headquarters staff, retiring as colonel. Previously he was with the company 15 years at Passaic.

General Detroit Corporation Opens Philadelphia Office

Elmer A. Marquardt has been appointed zone sales manager in charge of an office recently opened by The General Detroit Corporation and The General Pacific Corporation at 1427 North Broad Street, Philadelphia. As both office and warehouse, the new branch will serve the tri-state area surrounding the city.

E. A. Marquardt

De Laval Announces Aircraft Gas Turbine Development

One of the largest government-sponsored aircraft gas-turbine experimental projects in the country has been identified with De Laval Steam Turbine Company, Trenton, New Jersey.

H. L. Watson, president of the company, formally announced completion of plans for the project, which is being carried out by the recently organized De Laval Aircraft Gas Turbine Division.

While details of the new aircraft gas-turbine could not be made public it was disclosed that the project involves, among other features, a new and unusual concept of compressor and gas-turbine design.

The new aircraft gas-turbine designs are identified with Rudolph Birmann, consulting engineer, who for many years prior to the war was in charge of De Laval turbine design and, since 1937, has been prominently associated with research, design and manufacture of aircraft compressors and gas turbines.

Timken Steel and Tube Names Three Representatives

The Steel and Tube Division of The Timken Roller Bearing Company has appointed three district sales engineers: Sherman R. Lyle to the Cleveland district, William Earle Bryden to the Chicago district, and Alfred J. Kinnucan to the New York district.

Lyle graduated in 1938 from Case School of Applied Science with a bachelor of science degree in metallurgical engineering and received his Met. E. degree from the same institution in 1945. After two years in the metallurgical department of Carnegie-Illinois Steel Company in Farrell, Pennsylvania, he joined Timken in 1940 as a metallurgical assistant in the Steel and Tube Division. In 1945 he transferred to metallurgical service, and six months later to the sales department.

Bryden joined the Timken organization as an assistant in the metallurgical

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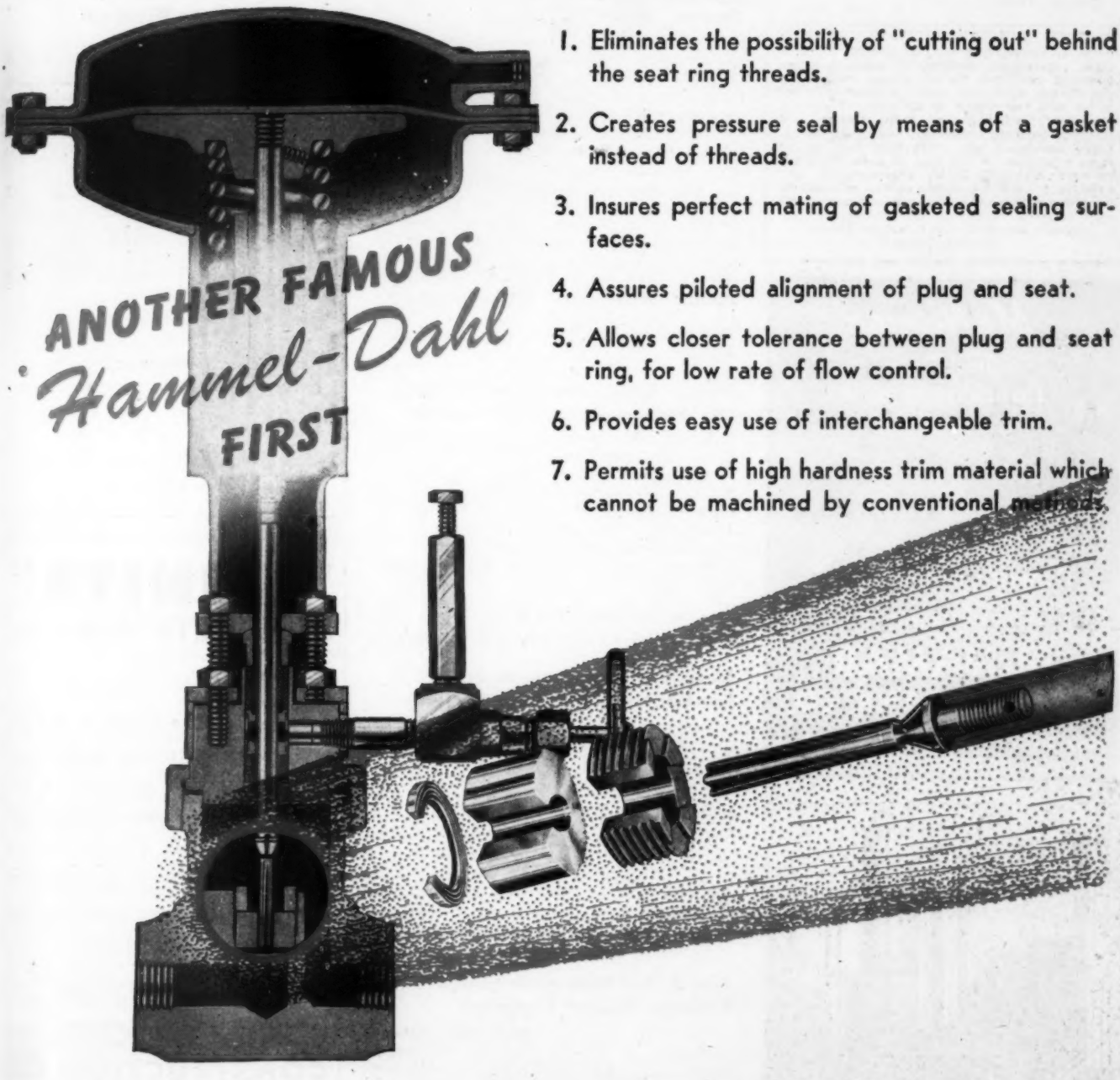

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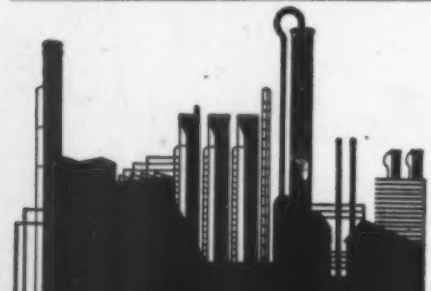

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department in 1937 and in 1940 became sales engineer for the New York district. During the war he served first with WPB and then three years in the Navy. He has a bachelor of science degree in metallurgy from Case School of Applied Science.

Kinnucan was on active duty with the Navy throughout the war. He graduated from Temple University in 1940, and went to work for Peter A. Frasses & Company, steel warehousing firm at Philadelphia. Later he joined Empire Finished Steel Corporation as district sales representative at Philadelphia.

McDonald Joins Pacific Pumps' New York Staff

R. C. McDonald has been added to the New York staff of Pacific Pumps, Inc. A graduate in chemical engineering from Northeastern University, Boston, he has had experience in engineering and selection of pumps and compressors, refinery and chemical plant installations with Worthington Pump and Machinery Corporation and Foster Wheeler Corporation.

R. C. McDonald

Meyer American Representative Oil Well Engineering Company

A. Robert Meyer has been appointed American representative of The Oil Well Engineering Company, Ltd. (London), and manager of the company's New York office. He succeeds E. T. Landgraff, who resigned recently to join Chiksan Company.

Meyer recently was discharged from the United States Army Air Force after 3½ years service, and previously was with National Supply Company at Toledo, Dallas and elsewhere for 14 years.

Newquist in Charge of Sales For Roots-Connersville

Ralph R. Newquist has been elected vice president in charge of sales of Roots-Connersville Blower Corporation, Connersville, Indiana. He is a graduate of Pennsylvania State College, and has been successively employed by Reliance Electric & Engineering Company, Louis Allis Company, and Allis-Chalmers Manufacturing Company. He is a member of AIEE, the Society of Naval Engineers, and American Society of Naval Architecture and Marine Engineering.

Erle J. Zoll Named President Chicago-Wilcox Company

Erle J. Zoll recently was named president of Chicago-Wilcox Manufacturing Company, Chicago. In addition to his new duties he will continue to be in charge of sales, as he has been for the past 29 years.

Weston Electrical Instrument Corporation Adding to Facilities

Weston Electrical Instrument Corporation is constructing an engineering and administration building on the plant grounds at Newark, New Jersey. The three-story structure will have 78,620 square feet of floor area.

Cordox Corporation Makes Two Appointments

Appointment of Felix C. Rodgers as general manager of its fire division and of H. V. Williamson as director of research has been announced by Cordox Corporation, Chicago.

For the past several years Rodgers has been a district manager for the fire division, with headquarters at Pittsburgh. As general manager he will be located at Chicago. Williamson formerly was chief engineer of the company's research division.

Broxham Sales Head for Graver Fabricated Plate Division

Willard M. Broxham has been named manager of sales for the fabricated plate division of Graver Tank & Manufacturing Company. He formerly was manager of the company's Philadelphia office, and now makes his headquarters at the general office in East Chicago, Indiana.

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